HEAVY METALS IN THE SEDIMENTS

OF AN ARCTIC LAGOON, NORTHERN ALASKA

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HEAVY METALS IN THE SEDIMENTS OF AN ARCTIC LAGOON, NORTHERN ALASKA

A

THESIS

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By

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ABSTRACT

The total abundances of V, Cr, Mn, Fe, Co, Ni, Cu, and Zn in oxic, nearshore sediments of north arctic Alaska, are similar to those of corresponding pristine or pre-industrial-age sediments of many tropical-temperate regions, and thus do not reflect the unique weathering and depositional processes of the Arctic.

Laboratory experiments on metal partitioning suggest that about 50% of the metal contents (<20% for V, Cr), which are bound predominantly in Fe oxides, may be mobilized into solution following the onset of reducing conditions at the sediment surface. A three-part extraction design would be effective in discriminating the three major associations of heavy metals in the sediments: easily leachable, Fe hydroxides, and refractory particles, and is recommended for use in monitoring the nearshore Beaufort Sea for future contamination by heavy metals.

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In closing, I must add that it has taken quite awhile for me to learn that the pursuit of knowledge is a human enterprise, and therefore is generally subject to the rules and regulations of society. I dedicate this closing of a period of my life to my late Grandfather, Walter R. Ward, and my Godson, Chad Michael Wells, between whose lives is found my own.

INTRODUCTION

Heavy Metals in Coastal Environments

Following the industrial revolution, municipalities and industries have been discharging a wide variety of potential pollutants in increasing amounts into coastal environments (Figure 1). In recent years, one group of these pollutants, heavy metals (e.g., mercury, copper, chromium), has been studied extensively along coastlines of the temperate belt. Dramatic instances of large influxes of heavy metals contained in industrial wastes, which have contaminated coastal food supplies resulting in acute or chronic health effects for local human populations (e.g., Minamata Bay, Japan), have been an important impetus to the recent surge of research. For over a decade, information has been rapidly accumulating on the sources, magnitudes, geochemical behaviors, and ecological effects of heavy-metal contaminants in coastal zones (Table 1). The marine environment is now an important topic when hundreds of scientists meet biennially at an international conference to discuss and attempt to synthesize recent findings concerning heavy metals in the environment (CEP Consultants Ltd, 1979-81-83).

It is now generally recognized that nearshore marine habitats may have limited capacities to absorb heavy-metal inputs without suffering ecological damage. There are at least six major sources of heavy metals in coastal environments (Forstner, 1980; Forstner & Wittmann, 1981, p. 30):

1) Products of geological weathering.
2) Wastes from the industrial processing of ores and metals.
3) Discharge of metals and metal compounds used by industries.
4) Burning of fossil fuels, production of cement and bricks.
5) Leaching of metals from garbage and solid-waste dumps.
6) Animal and human excretions.

In appreciation of the possible deleterious effects of these kinds of heavy-metal inputs to estuarine and nearshore ecosystems, civil and international authorities have begun to take action in controlling the kinds and rates of discharges and dumpings of waste materials (WHO-UNEP, 1982). Steps are also being made to minimize the impacts to marine ecosystems which are already contaminated (Peterson & Randolph, 1977).

Importance of Sediments on the Fate of Heavy Metals in the Nearshore

Estuarine and coastal environments, where terrestrial and marine waters meet, are very dynamic in physical, chemical, and biological processes. Heavy metals can be

exchanged between the aqueous, particulate, biotic and atmospheric compartments (Duinker et al., 1974; Leckie & James, 1975; Olsen et al., 1982). Quantitatively, the most important component in the nearshore environment, with respect to the cycling of heavy metals, is sediment. Sediment consists of the suspended and accumulated particles of terrigenous and marine origin, including transported weathering products, chemical precipitates, and biogenic residues.

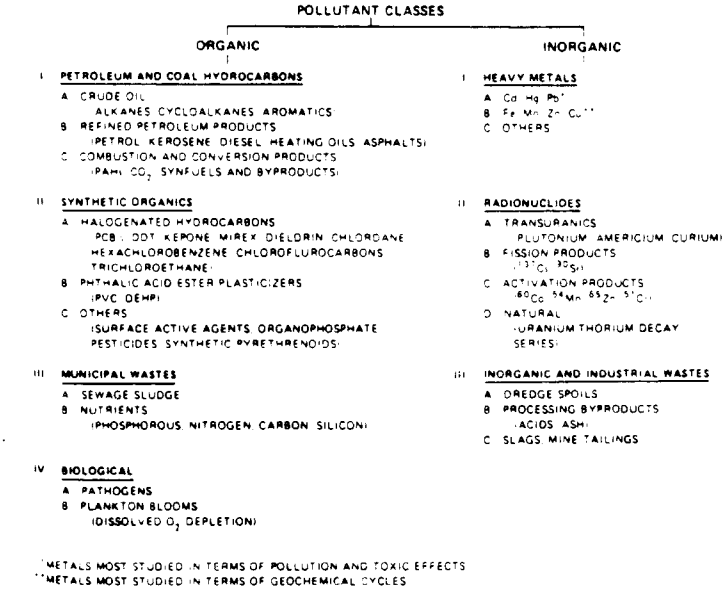

Figure 1. Classes of known or potential pollutants in the coastal zone (reprinted from Olsen et al., 1982).

In unpolluted areas, the bulk of heavy metals entering the nearshore are already associated with suspended

particles, either adsorbed to their charged surfaces or contained within their structures (Troup & Bricker, 1975; Gibbs, 1977; Martin & Maybeck, 1979; Bourg, 1981; Etcheber et al., 1981; Yeats & Bewer, 1982). At the freshwater-seawater interface, trace metals present in the dissolved or colloidal phase are rapidly sequestered by flocculating aggregates of iron-oxide sols and organic colloids (Sholkovitz, 1978). Thus, a major portion of the heavy metals which enter estuarine or coastal waters are removed to the sediment (Duinker & Nolting, 1977; Turekian, 1977; Duinker, 1980). The study of the sediment component therefore is important in monitoring the input and cycling of heavy metals in the nearshore (Figure 2).

Case Studies of the Accumulation of Heavy Metals in Nearshore Sediments

It has been observed that the concentrations of some heavy metals are several times background levels in sediments adjacent to the submarine discharge points of domestic and industrial wastes (Heltz et al., 1975; Papakostidis et al., 1975; Amiel & Navrot, 1978; Hershelman et al., 1981). Concentrations typically decrease along gradients leading away from the release points of the wastewater outfalls. Sediments in estuaries also trap resistant ore minerals or mobilized ore elements transported by rivers from the mining areas (Yim et al.,

1976). This particle "conspiracy" is also indicated by laboratory experiments with radiotracers (Aston & Duursma, 1973) and field studies of radioisotopes discharged into shallow marine environments (Olsen, et al., 1980).

TABLE 1

MAJOR AREAS OF RESEARCH ON
HEAVY METALS IN COASTAL ENVIRONMENTS

Area of Research	Reference
Sources, magnitudes, geochemical pathways, fates, and ecological effects of heavy metals in:	
Southern California	Katz & Kaplan, 1981 Bascom, 1982
World-wide	Erlenkeuser et al., 1974 Bryan, 1976a Goldberg, 1976 Burton & Young, 1980 Lyons & Fitzgerald, 1980 Baker, 1980 Forstner & Wittmann, 1981 Geyer, 1981
Environmental parameters and biological mechanisms affecting heavy-metal uptake and toxicity to marine organisms:	Bryan, 1976b Engel et al., 1981 Fowler et al., 1981 Luoma & Bryan, 1982 Jenne & Wildung, 1983
Use of biological indicator organisms to monitor heavy-metal contamination of coastal environments:	Phillips, 1977 Natl. Acad. Sci., 1980 Packer et al., 1980

Products of geological weathering, however, may sometimes be more important than anthropogenic sources for anomalous heavy-metal concentrations in nearshore sediments. For example, it was suggested that onshore deposits containing oolitic iron-stones and alum shales were more important than industrial inputs for the occurrence of elevated concentrations of iron, manganese, nickel, copper, and zinc in some coastal sediments of England (Taylor, 1979). Additionally, the source for high levels of chromium and nickel in nearshore sediments may sometimes be the erosion of onshore deposits of ultrabasic and basic rocks (Cosma et al., 1979).

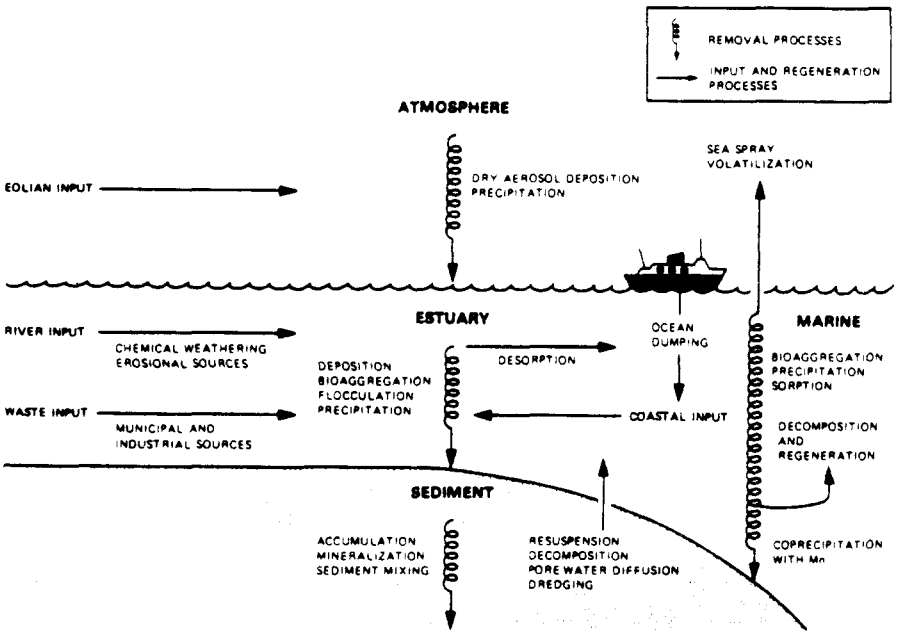

Figure 2. Biogeochemical processes affecting particle-pollutant dynamics in coastal marine environments (reprinted from Olsen et al., 1982).

Chemical Associations of Heavy Metals in Nearshore Sediments

Nearshore sediments are a very complex assemblage of inorganic and organic compounds of various compositions and stabilities. The principal components of sediment particles in nearshore oxic environments are silicate minerals, hydrous iron and manganese oxides, organic residues of plants and animals, and carbonate minerals. Heavy metals may become associated with these sediment constituents in several ways, including adsorption to surfaces, complexation, co-precipitation, and substitution for major elements in the lattice sites of detrital minerals (Figure 3).

It is important to ascertain the partitioning of heavy metals between these chemical phases in order to assess or predict the "available fractions" in the sediment. These are the metal phases potentially available to organisms, or susceptible to remobilization into the overlying water in response to perturbations in the physicochemical environment (Jenne & Luoma, 1977). For polluted sediments, this distinction is important for assessing ecological damage and the possibility of a hazardous accumulation of heavy metals by marine food items of humans. For pristine sediments, information on geochemical partitioning provides insight into the cycling of elements

in different environments. Data on total metal concentrations, however, only provide contrasts between environments, without any clues to the reasons.

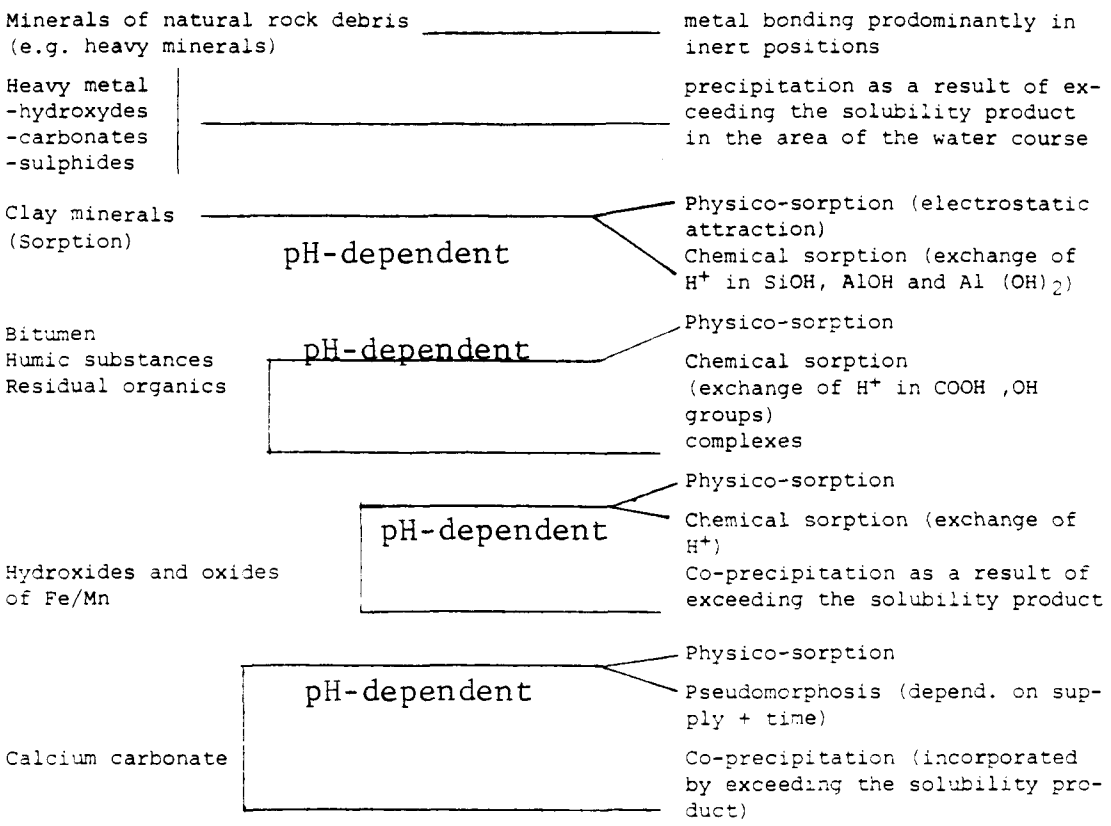

Figure 3. Carrier substances of heavy metals in natural particulates, and their corresponding bonding mechanisms (reprinted from Forstner et al., 1978).

Wet-chemical methods have been developed recently to "selectively" leach metals associated with the different geochemical phases in sediments. Treatments with ap-

propriate reagents are adopted, designed to mobilize the exchangeable components, dissolve carbonates, oxidize organic matter, reduce oxides, and so forth. These methods are useful in discriminating the weakly-bound metals from those incorporated in more stable phases by sequentially subjecting the sediment to reagents of increasing reactivity. Pollutant heavy metals generally become incorporated in the "available fractions" of sediments, by the processes of adsorption, complexation, or precipitation (Agemian & Chau, 1977; Chester & Voutsinou, 1981). Sequential extraction methods therefore are more specific than total analysis in establishing the heavy-metal contamination of sediments.

Heavy Metals in Arctic Nearshore Sediments

The focus for previous studies on the geochemistry of heavy metals in nearshore sediments has been the low and middle latitudes, nearby the major industrial and population centers of the world. At the time of this writing, I know of only a handful of useful investigations written in English, on heavy metals in arctic nearshore sediments outside the Alaskan Beaufort Sea. These are mostly from the Canadian Arctic (Table 2).

During the last decade, there has been a great surge in industrial activity, as related to petroleum explora-

tion and exploitation, in the Alaskan Arctic. The immediate and long-term potential for heavy-metal pollution arising from this ongoing development was a significant impetus for the present study. It was also an opportunity to contribute to the understanding of the geochemical cycles of metals in ice-stressed nearshore environments and along passive continental margins, such as the Beaufort Sea.

Location and Heavy Metals Selected for Study

Simpson Lagoon, in North Arctic Alaska, was chosen for the present study because of logistical conveniences, an already established and growing data base of various ecological and physical parameters (e.g., Kinney et al., 1972; Tucker, 1973; Alexander et al., 1975; Tucker & Burrell, 1977; Johnson & J. W. Richardson, 1981), and its critical location adjacent to proposed offshore oil and gas lease sales (U.S. Dept. Interior, 1982). Simpson Lagoon is also a biologically sensitive habitat and an historical and potential recreational resource (Johnson & J. W. Richardson, 1981; U.S. Dept. Interior, 1982).

Several criteria were used to select the metals to be examined: vanadium (V), chromium (Cr), manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), copper (Cu), and zinc (Zn). First, one or more of these elements are usually

concentrated in domestic and industrial effluents which contaminate many coastal sediments around the world (Forstner & Wittmann, 1981). The resulting environmental concern has sparked the generation of a very large body of data on these heavy metals (e.g., Table 1), providing a basis for evaluating nearshore sediments of the Arctic. Further, these elements are relatively easy to analyze accurately with atomic absorption techniques, as compared to metals such as mercury, arsenic, lead and cadmium. The establishment of baseline levels for heavy metals in Simpson Lagoon sediments was expedited by first acquiring data for the more easily analyzed metals.

Secondly, Fe and Mn are included because of their environmental and geochemical importance. Iron is generally over two orders of magnitude greater in abundance than the other (trace) metals in nearshore sediments; its hydrous-oxide state is believed to be an important scavenger (and thus a sink) for a number of trace metals in the environment (Goldberg, 1954; Krauskopf, 1956; Jenne, 1968; Lee, 1975). The relatively less abundant Mn is also a sink for many trace metals, but it is also a useful indicator of sedimentary redox conditions (Graham et al., 1976; Yeats et al., 1979; Wilke and Dayal, 1982). The other six trace metals are essential micronutrients for many marine organisms, but become toxic

at elevated concentrations (Bryan, 1976a; Wood, 1975; Wood et al., 1975).

In addition to the eight metals, grain-size distributions, clay mineralogy, and carbonate ($CO_3^{=}$) and organic-carbon (OC) contents of sediments were determined to aid in understanding the heavy-metal distributions in the sediment.

Importance of This Study

In regions of the Earth remote from the industrialized belts, the environment may be considered relatively pristine, providing an opportunity for research on the natural geochemical cycles of elements. The present study in the remote Alaskan Arctic establishes valuable baselines for the natural levels of heavy metals in the nearshore sediments. This data can be used to monitor the coastal zone for contaminant impacts from the ongoing activities of the petroleum and related industries in the adjacent onshore and offshore areas.

This study most likely represents the first effort in the nearshore arctic of Alaska to acquire an extensive geochemical data set for sediments from a relatively small area. It is also probably the first elucidation of the partitioning of heavy metals in nearshore arctic sediments using sequential extraction methodology.

TABLE 2

SOME STUDIES OF HEAVY METALS IN ARCTIC NEARSHORE SEDIMENTS OUTSIDE OF ALASKA

Reference	Location	Metals Studied	Comments
Macdougall & Harriss, 1967	Canadian Arctic Archipelago, 77°N Lat.	V, Cr, Mn, Fe, Co, Ni, Cu	<63μm, Total Digest
Beak Consultants Ltd, 1978	Mackenzie Delta & Estuary, 69°N Lat.	Hg, As, Cr, Cd, Pb, Zn	Total sediment, Hot acid extraction
Campbell & Loring, 1980	Baffin Bay & adjacent waters, 65°to 78° N Lat.	Zn, Cu, Pb, Co, Ni, V, Cr, Hg, Mn	Total sediment, Total Digest
Kravitz, 1982	Kane Basin, Greenland	Fe, Mn, Cr, Co, Ni, Zn, Cu, Ti	<2μm, Total Fusion
Wright, 1974a, 1974b	Barents Sea	Ti, Fe, Mn, Zn, Ni, Cu, As, Pb	Total, silt, clay fractions, X-ray flourescence

The recent improvements in accuracy and detection limits of analytical techniques (e.g., atomic absorption spectrophotometry), and the rapid accumulation of extensive world-wide data, have now made it imperative to revise the average abundances of heavy metals in nearshore

sediments, as compiled by Turekian & Wedepohl (1961), among others. It is important that the new data from arctic localities now becoming available be included in the new estimates, in light of the relatively wide continental shelves of the Arctic which have not previously contributed to the world-wide averages.

Previous Studies of Heavy Metals in the Sediments of the Alaskan Beaufort Sea

A modest amount of information on heavy metals in sediments of the Alaskan Beaufort Sea is already available. This includes (Table 3) broad regional surveys of the continental shelf (Naidu & Hood, 1972; Weiss et al., 1974; Naidu, 1975), of the inner and outer shelf areas (Barnes, 1974; Barnes et al., 1974), and of the inner shelf (Naidu & Mowatt, 1974). Prudhoe Bay and its adjacent shallow waters have received special attention (Naidu, 1976a, 1976b; Grider et al., 1977; Northern Technical Services, 1981), as well as a potential oil company drilling site in shallow water just west of Harrison Bay (Toimil & England, 1983). These studies have yielded heavy-metal data for seven sampling stations in Simpson Lagoon (Barnes, 1974; Barnes et al., 1974; Naidu & Mowatt, 1974).

TABLE 3

PREVIOUS STUDIES OF HEAVY METALS IN SEDIMENTS OF THE ALASKAN BEAUFORT SEA

Reference	Location	Metals Studied	Comments
Naidu & Hood, 1972	Shelf between Pt. Barrow & Prudhoe Bay	Fe , Mn, Zn, Ni, Co, Cu	<2mm, Total digest
Barnes, 1974	Inner & Outer shelf	Hg, Cu, Pb, Zn, As	<2mm, Leachable
Barnes et al., 1974	Inner & outer shelf	Cu, Pb, Zn, Hg, As	<2mm, Leachable
Naidu & Mowatt, 1974	Inner shelf, between Cape Halkett & the Canning River	Fe, Mn, Cu, Co	Total sediment, Total digest
Weiss et al., 1974	Sag., Colville Rivers; outer shelf	Hg	Total sediment, Hot digest
Naidu, 1975	Outer shelf between Cape Simpson & Canning R.	Fe, Mn, Cu, Ni, Zn	<2mm, Total digest
Naidu, 1976a,b	Prudhoe Bay dock	Ni, V, Cr, Cu	Total sediment, Total digest
Grider et al., 1977	Prudhoe Bay	Ni, Zn, Pb Cd, Cr, Fe, Cu, V	Total sediment, Carbonate fusion

TABLE 3

(continued)

Reference	Location	Metals Studied	Comments
Northern Technical Services, 1981	North of Prudhoe Bay	Co, Cr, Cu, Fe, Mn, Ni, Pb, V, Zn	Total sediment, Total digest
Toimil & England, 1983	Just west of Harrison Bay	Co, Cr, Cu, Fe, Mn, Ni, Pb, V, Zn	Total sediment, Hot digest

These baseline data are essentially analyses of total or leachable contents of a suite of metals. There have been, however, no previous attempts to my knowledge, to ascertain in detail the geochemical fractionation of the metals in the sediments of the inner and outer shelf areas of the Alaskan Beaufort Sea.

Specific Objectives of This Study

1 To determine the total abundances of V, Cr, Mn, Fe, Co, Ni, Cu, and Zn in lagoon sediments of North Arctic Alaska.

2 To assess the relative abundances of the suite of heavy metals in some operationally-defined phases of the lagoonal sediments.

3 To gain an insight into the role of some of the lithological attributes of the sediments (texture, clay mineralogy, and carbonate and organic-carbon contents), on the abundances and partitioning of heavy metals in the lagoonal sediments.

4 To critically appraise the precision of sample collection, and the precision and accuracy of analysis, for estimates of the total and operationally-defined fractions of the suite of heavy metals in the lagoonal sediments.

5 To compare the total abundances of the eight metals in nearshore sediments of North Arctic Alaska with their abundances in the nearshore sediments of tropical and temperate regions of the world.

6 To attempt to predict the effects of some potential and ongoing industrial activities in the Alaskan Arctic, on the abundances and fates of heavy metals in the nearshore sediments, based on estimates of their fractionation patterns obtained in this study.

Geographic Setting

The geographic region of relevance to this study, northern Alaska, lies completely within the Arctic Circle, between degrees 68 and 71 north latitude (Figure 4).

The onset of the present cold or frost climate of northern Alaska was initiated following the world-wide cooling in Late Pliocene, about two to three million years ago (Steuerwald et al., 1968; Nekrasov, 1983; Popov, 1983). During this period, the coastal plain of Arctic Alaska was exposed to several cycles of marine transgressions and regressions, and glacial ice extended and

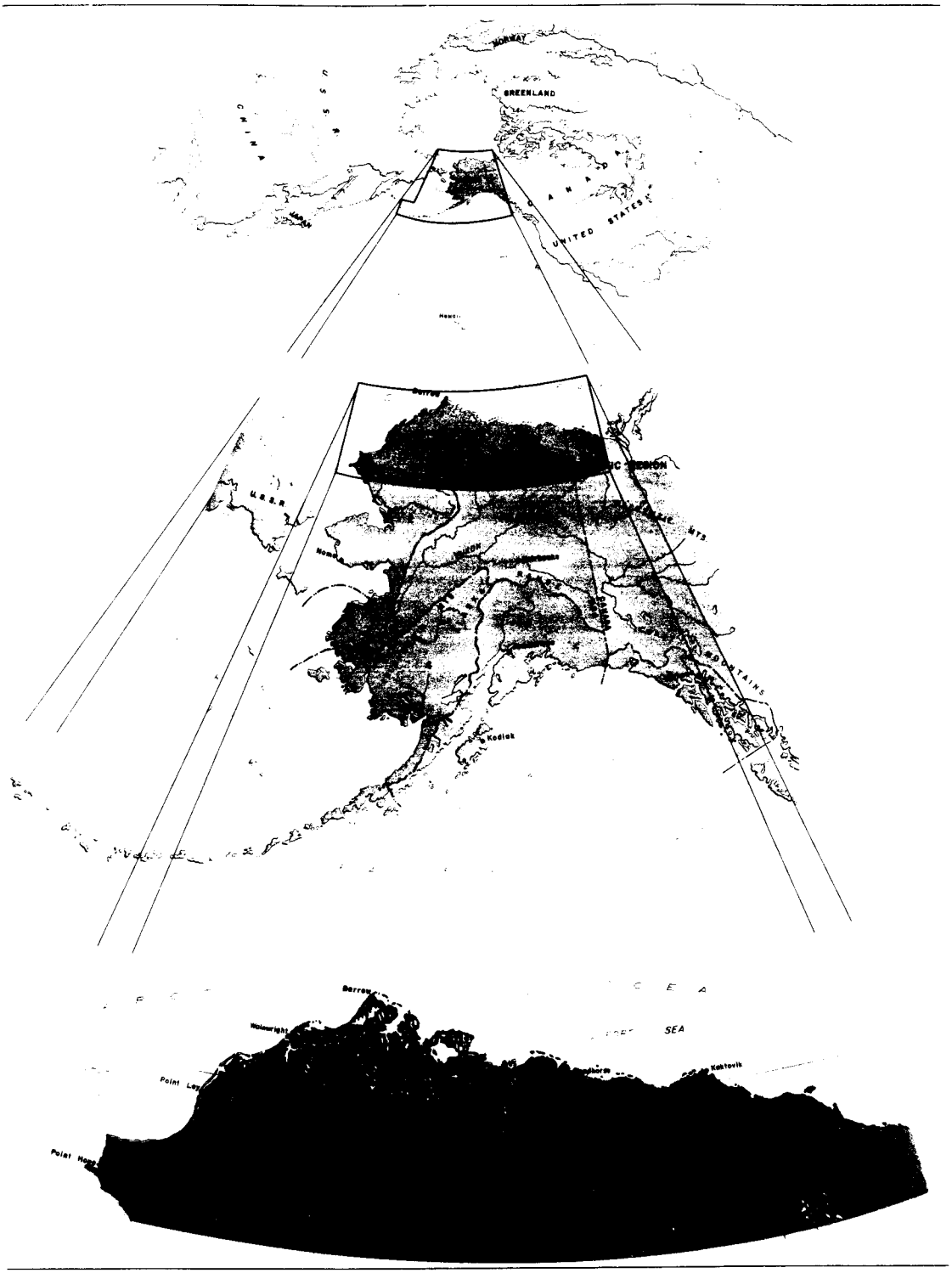

Figure 4. Location of northern Alaska (reprinted from Selkregg, 1975).

retreated within the adjacent Brooks Range, coinciding with the glacial-interglacial episodes of the Pleistocene Epoch (McCulloch, 1967; Brigham and Miller, 1983).

Most of the coastal plain is underlain by up to 600 meters of permafrost (Osterkamp & Payne, 1981): probably a relict of Wisconsin Stade (Embleton and King, 1975). The present tundra surface probably originates from the last low stand of the sea, about 18 thousand years ago (Hopkins, 1967, 1982), and contains typical periglacial features such as thaw lakes and ice-wedge polygons. Surficial deposits are predominantly reworked marine sands, silts and gravels of the Quaternary Gubik Formation (Black, 1964), which overlies Cretaceous and Tertiary deposits west and east of the Colville River respectively. Air temperatures are relatively cold year round (-40° C to +10° C), and precipitation is less than 15 cm/yr, so that the coastal plain may be considered a semi-arid region.

The pedogenic process in the cold and dry climate of the Alaskan Arctic is characterized by the retardation of chemical reactions, resulting in the preservation of unstable minerals like feldspars, and a general lack of formation of clay minerals (Hill, 1957; Kunze et al., 1968; Naidu & Mowatt, 1983). The latter are mostly reworked products from sedimentary rocks principally of the Brooks Range (Tedrow, 1977). Under the frigid

climatic conditions, low annual erosion and transport rates prevail (Popov, 1983). Therefore, terrigenous debris mostly constituted of mechanically reduced particles is transported to the coastal region. In short, throughout the Quaternary Period, the process of denudation of the landscape of northern Alaska has been quite unique by comparison with the more temperate regions of the world (Birot, 1968; Lukashev, 1970; Peltier, 1973).

AREA OF STUDY & DEPOSITIONAL SETTING

Introduction

Simpson Lagoon is located near the midpoint of the Beaufort Sea coast of Alaska, between the Colville River Delta and Prudhoe Bay (Figures 5-7). Stratigraphic studies suggest that the lagoon evolved from the tundra- and lake-covered coastal plain 4500 years ago (Naidu et al., 1984). Relict coastal highlands, now represented by Pingok Island, separate Simpson Lagoon from the open Beaufort Sea. At least 70 cm of Holocene sediments have accumulated in central Simpson Lagoon (Naidu et al., 1982, 1984).

Source of the Sediments in Simpson Lagoon

For an understanding of the distribution of elements in nearshore arctic sediments, an appreciation of the unique sedimentary processes in the region is important. The sediments accumulating on the floor of Simpson Lagoon are supplied by a variety of sources and mode of transport; ice plays a significant role.

North Slope rivers are the most important source of terrigenous debris to the nearshore zone and may discharge

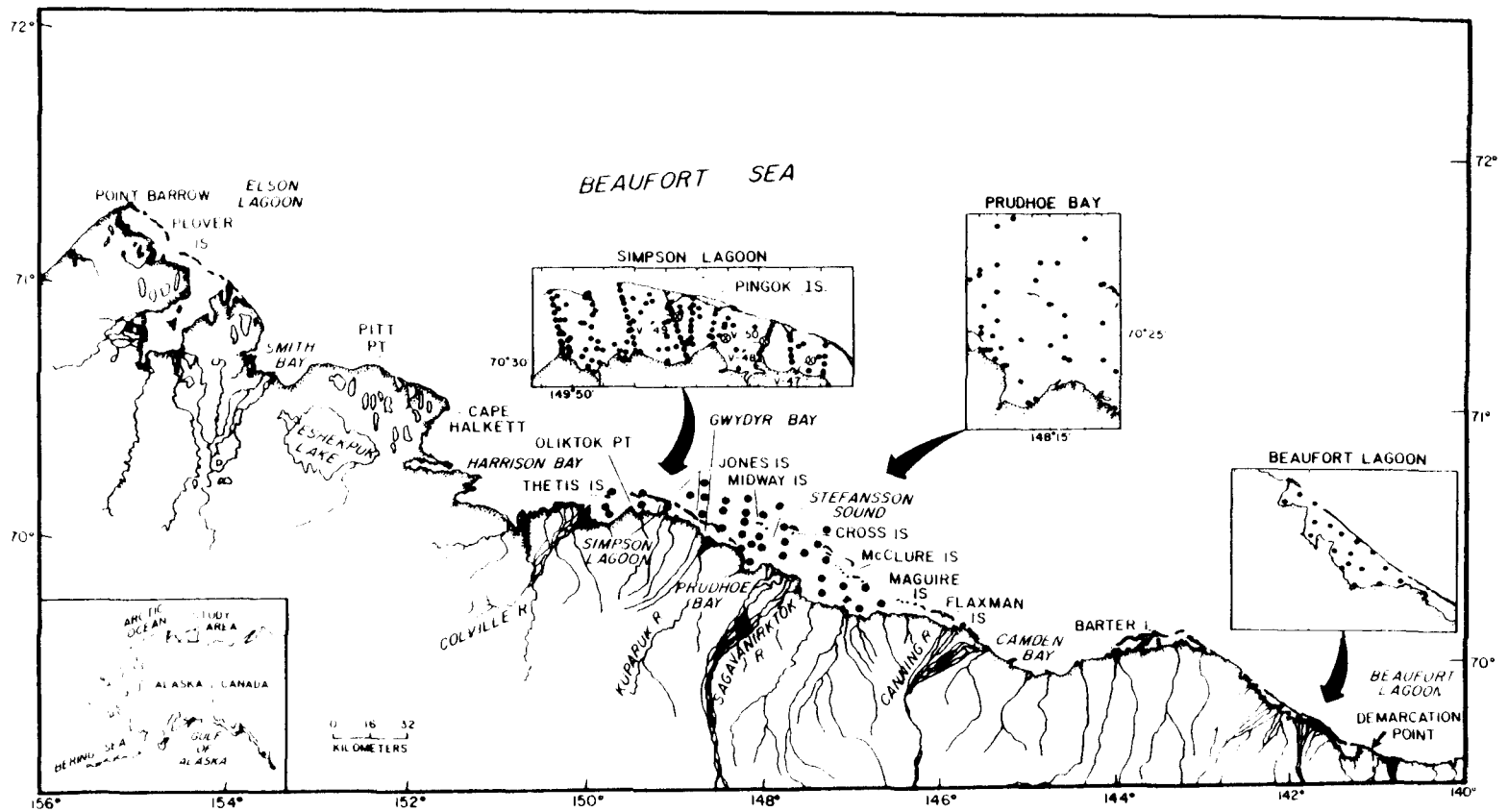

Figure 5. Location of Simpson Lagoon along the Beaufort Sea coast of Alaska.

76% of their annual load during the first two weeks of the breakup period, which occurs around June 15 (Arnborg et al., 1967; Drage et al., 1983). The two most important rivers supplying detritus to Simpson Lagoon are the Colville and Kuparuk (Figures 5-7). In addition, there are 5 minor streams which drain the coastal plain between these two major rivers (Drage et al., 1983), but their combined sediment load is by comparison very small. At spring breakup, most of the fluvial detritus is initially deposited on the shorefast ice situated off the river mouths. Much of this terrestrial debris subsequently drains into the water column through cracks and channels in the sea ice (Walker, 1974), and may be transported in suspension into Simpson Lagoon by the advancing flood waters from the rivers (Barnes & Reimnitz, 1972; Reimnitz & Bruder, 1972; Walker, 1973; Cannon et al., 1978).

The secondary sources for sediments in Simpson Lagoon are the coastal bluffs and beaches along the adjacent mainland and barrier islands. The volume of sediment supplied by the thermal erosion of these sources is estimated to be an order of magnitude less than that being delivered by the rivers (Cannon et al., 1978).

In summer, fine-grained sediments resuspended periodically by wave and current agitation, are transported westward (Naidu & Mowatt, 1983) by longshore cur-

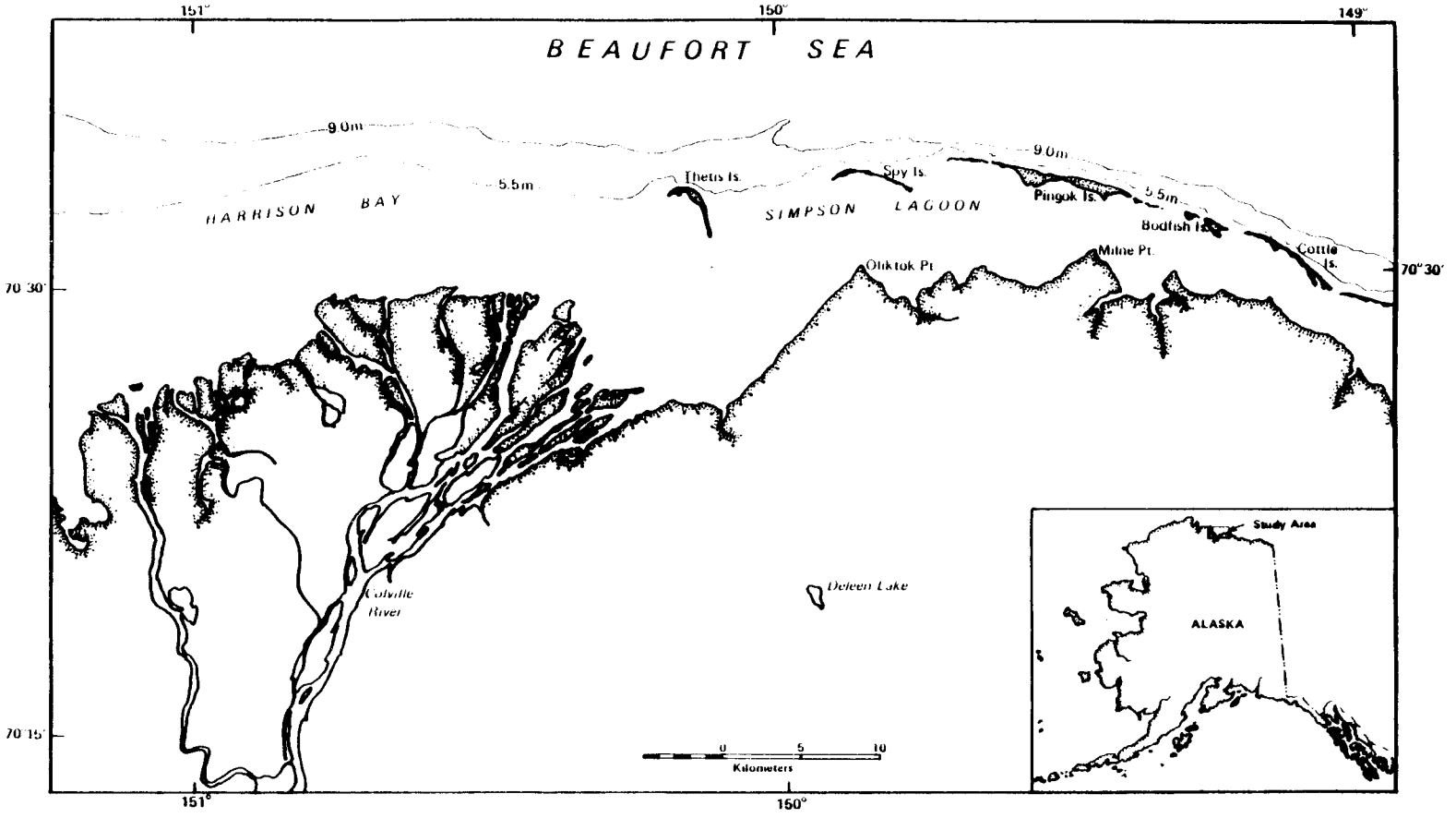

Figure 6. Location of Simpson Lagoon relative to the Colville River Delta.

rents (Matthews, 1981). It seems likely then, that the central axis (> 2 m depth) of Simpson Lagoon receives the bulk of its sediment from the Kuparuk River to the east, and possibly a significant contribution from the Sagavanirktok (Naidu et al., 1982). In contrast, most of the sand and gravel concentrated along the northern and southern margins of Simpson Lagoon are very likely lag deposits derived locally from the coastal bluffs (Naidu et al., 1982). Some coarse particles, however, are redistributed to the central lagoon by storm surges, longshore currents, or ice rafting. Tentative results indicate the rate of accumulation of sediment in the lagoon is in the order of 0.5 cm/year (Naidu, 1981), which appears to be relatively low for a deltaic area, possibly due to the low annual rate of denudation of the hinterland and short season in which sedimentary processes can operate (Derbyshire, 1976).

Sediment Dynamics

Simpson Lagoon is generally less than three meters deep, with 65% of the area (central portion) deeper than 1.8 meters (Johnson & W. J. Richardson, 1981, p. 123). Sediments in such an extremely shallow basin undoubtedly are subject to reworking and redistribution processes. For example, river floodwaters which overflow the intact

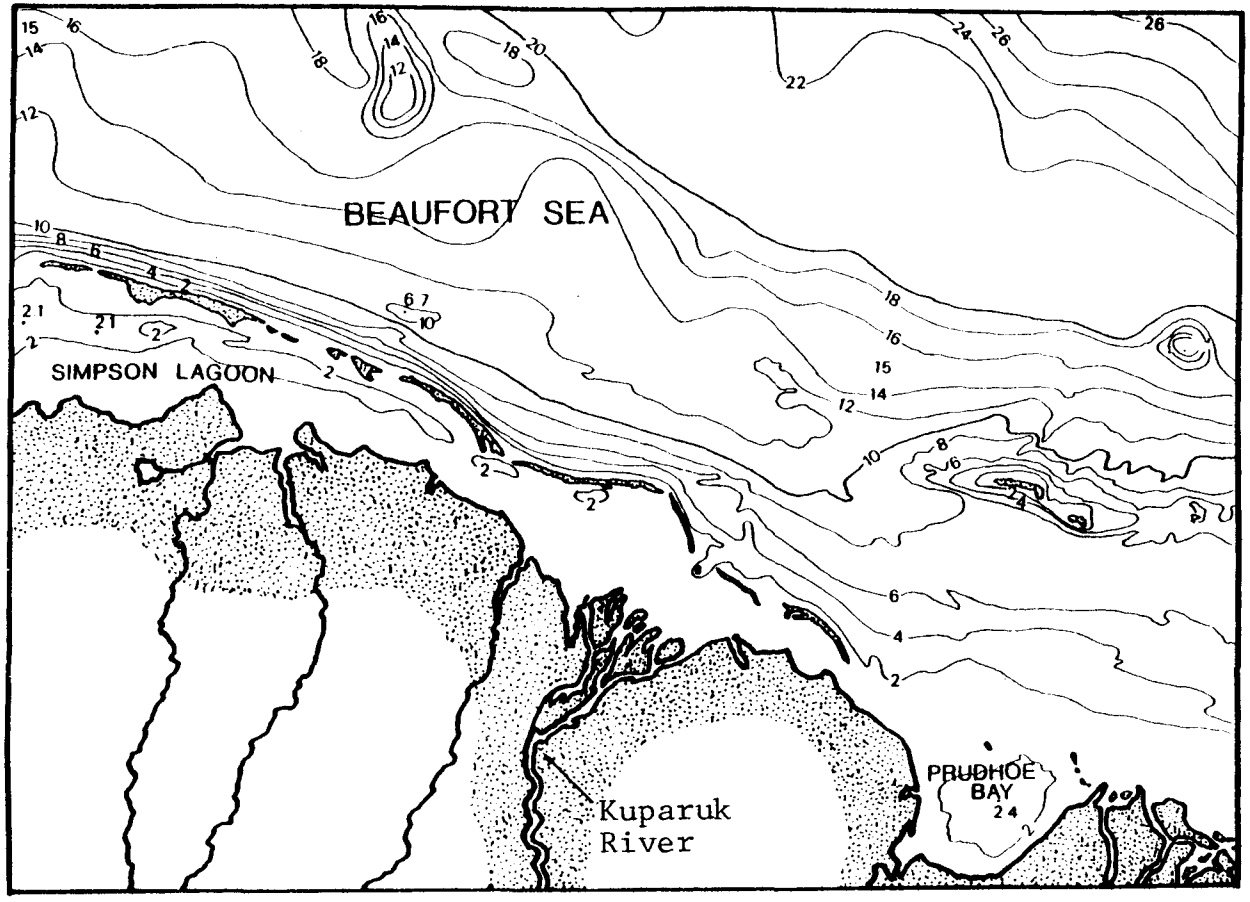

Figure 7. Location of Simpson Lagoon relative to the Kuparuk River, and Prudhoe Bay (reprinted from Johnson & J. W. Richardson, 1981, Vol. 8, p. 305).

lagoon ice in early spring, drain through cracks with such intensity that locally they excavate circular depressions up to four meters deep into the sediments (Reimnitz and Kempema, 1982). During spring breakup and fall freeze-up, the landfast ice may scour the beach and very shallow areas (Weeks, 1980; Weeks et al., 1983). Sediment frozen to the underside of bottomfast ice (up to 2 m thick) by late winter is ice rafted during the spring event (Barnes & Reimnitz, 1974; Barnes et al., 1982). In contrast to the open shelf, however, offshore shoals and the barrier islands generally protect the deeper areas (> 2 m) of Simpson Lagoon from the plowing of its bottom by grounded ice (Reimnitz et al., 1978). By late winter, constriction of flow below the ice canopy during the late stages of ice growth is likely to result in intensified currents and the winnowing and transport of sediment beyond the two-meter isobath (Barnes & Reimnitz, 1973, 1974).

In summer, too, wave and current agitation in the shallow waters of the lagoon are effective in resuspending the sediments. Of major importance to long-term sediment transport are the cataclysmic effects of occasional storms during the open-water season, which may cause twenty years of normal sediment transport in a few hours (Hume and Schalk, 1967). The mean spring tidal range is only about 15 cm (Matthews, 1983); only in late winter, when con-

stricted flow intensifies their action, are tidal currents capable of sediment redistribution.

Sediment dynamics within shallow lagoons of the Arctic are poorly understood (Toimil & Reimnitz, 1979), but the reworking of sediment by ice and shallow water-related processes would seem to maintain well-aerated surficial sediments year-round.

Chemical Effects From Brine Exclusion During Freezing of Sea Ice

Where ice approaches and may contact the bottom by late winter in Simpson Lagoon (inside 2 m isobath), the surficial sediments most probably refreeze every year. At the sediment-ice interface, cold, dense brine, excluded from the lagoon ice, may penetrate into the sediment pores, causing chemical exchange between the brine and the interstitial waters. Occasionally, the brines are trapped in restricted pockets of water, and salinities over 50°/$_{oo}$ are common during the month of May (Schell & Hall, 1972; Aagaard, 1978). The saline wave front may travel up to one to two meters down the sediment column, displacing solutes ahead of it (Osterkamp, 1975; Page & Iskandar, 1978; Harrison & Osterkamp, 1982). The fate of any metals thus mobilized from the adsorbed/exchangeable phases of sediments are not known. Additionally, spring breakup is a unique period for the lagoon waters. As the river

floodwaters drain through the ice, they flush the hypersaline water (40‰-50‰) which had built up over winter (Matthews & Stringer, 1984).

Thus, sediments of Simpson Lagoon are exposed to both rapid and seasonal extremes of physical disturbance and water chemistry. Consequently, in the ice-stressed lagoons, it would seem that heavy metals have complex geochemical cycles.

MATERIALS AND METHODS

Sediment Collection and Storage

Five north-south transects using a Zodiac were made in August, 1977 across the western half of Simpson Lagoon (about 115 km^2). The top 15 cm (about two cubic decimeters, dm^3) of the sediments at 39 locations (Figure 8), were retrieved using a hand-operated, Ekman grab sampler. Replicate grabs (three to five) were taken at five of these stations (Appendix I), to estimate local variations of the measured parameters (intra-station variance). After collection, the grab samples were retained in polyethylene bags or plastic boxes at about 5°C on Pingok Island, prior to their dispatch to the Fairbanks laboratory. In the laboratory, they were stored frozen until ready for analysis. The storage of samples at low temperatures was adopted to minimize the modification of the original fractionation states of the metals, by preventing organic decomposition and biochemical reactions.

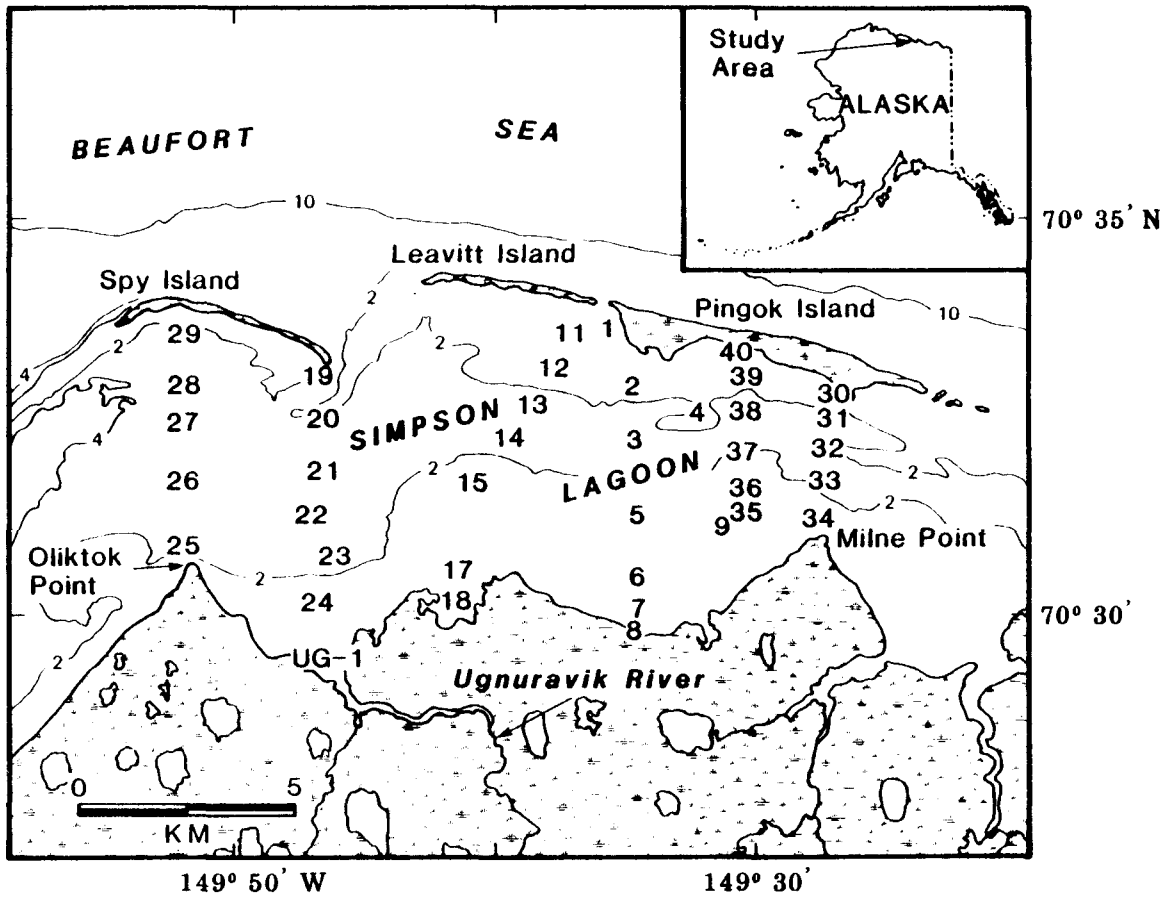

Figure 8. Locations in western Simpson Lagoon where sediment samples were retrieved. Samples taken approximately at the center of each bold number. Depth contours in meters.

Estimation of Grain-Size Parameters

In the laboratory, the sediment samples were thawed, thoroughly mixed, and two, approximately 50-gm splits (dry wt) were taken (Figure 9): one for grain-size analysis, the other for clay-mineral determinations (next section). Grain-size fractions were obtained by the conventional methods of sieving the sand and gravel, and pipetting the silt and clay (Krumbein & Pettijohn, 1938; Folk, 1974; Friedman & Johnson, 1982). Standard grain-size parameters (formulae after Folk & Ward, 1957), were then calculated from the fraction weights by the University of Washington SEDAN program, using the University of Alaska Honeywell 66/40 computer.

Determining the Relative Proportions of Clay Minerals

Clay-mineral analysis was undertaken in an attempt to ascertain if any relationships exist between these minerals and the heavy-metal distributions in the sediments of Simpson Lagoon. Clay-mineral separation and identifications were accomplished following the procedures outlined in Naidu et al. (1971) and Naidu & Mowatt (1974). Briefly, the mud fractions (<62μm) of 50-gm splits (Figure 9), were treated with 30% hydrogen peroxide at 25°C, to destroy organic matter. The mud residues were then

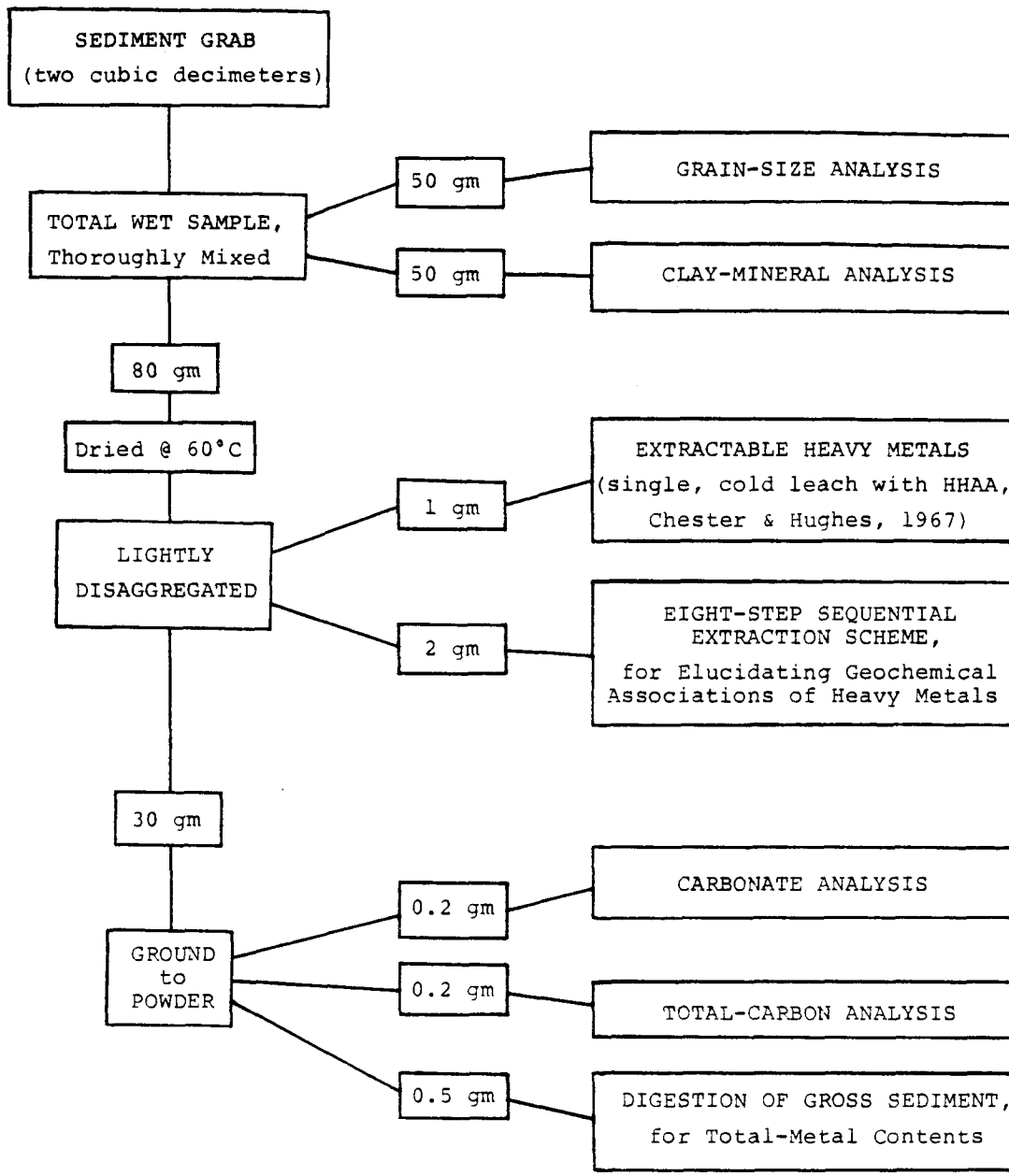

Figure 9. Flow diagram of sample splitting for each type of analysis in this study. Dry weights depicted throughout. Details of analytical methods discussed in "methods" section.

dispersed in distilled water, and the <2μm e.s.d. (equivalent spherical diameter) particles were separated by settling technique, according to Stokes' equation of settling under gravity (Wills, 1981). Following centrifugation, thick slurries of portions of the <2μm fractions, were smeared onto glass slides to obtain oriented grain mounts (Gibbs, 1965). Clay-mineral identifications were performed with a Phillips-Norelco X-ray diffractometer equipped with a scintillation detector, using nickel-filtered copper Kα radiation.

Clay minerals that expanded upon glycolation and gave a basal (001) diffraction peak in the neighborhood of 17 Å were assigned to the Expandable Group. For convenience, it may be assumed that the expandable minerals here defined belong mostly to the Smectite Group of minerals. Chlorite and kaolinite were differentiated by resolving the 3.5 Å doublet under slow scans (Biscaye, 1964). Semi-quantitative estimations of the relative proportions of the clay minerals were based on the method of Biscaye (1965).

Sample Preparation for the Analysis of Chemical Parameters

Additional splits of about 80 gm (dry wt) were taken from each grab sample for analysis of $CO_3^{=}$, OC, and the eight metals (Figure 9). The splits were dried at 60°C,

and then lightly disaggregated using a porcelain mortar and pestle. Approximately 50-gm portions of these coarse disaggregates were stored in acid-rinsed glass vials, for later use in the partial-extraction studies of heavy metals. It is believed that gentle disaggregation preserves the integrity of the minerals and the surface area of the sediment particles. The remaining quantities of the coarse separates were pulverized to fine powders with an agate mortar and pestle (Figure 9), and also stored in vials.

Determination of Carbonate and Organic Carbon

Total carbon and carbonate contents of the sediments were measured on separate, 0.2 gm portions of the fine powders (Figure 9), by a LECO TC-12 automatic carbon determinator (induction furnace), and a manometric technique (Hulsemann, 1966), respectively. The difference between the total- and carbonate-carbon estimates was defined as the organic carbon. The LECO instrument was calibrated using high and low carbon, steel-ring standards furnished by the LECO corporation. Dextrose (40% carbon by weight) was used as an internal standard. Calcium carbonate powder (100%) was used as the internal standard in the carbonate determinations.

Liberation of Heavy Metals From the Sediments

(a) background and rationale for the adopted methods

Physical, chemical and mineralogical studies of soils have employed chemical treatments for well over 50 years (e.g., Black, 1965; Jackson, 1974; Page, 1982). These "classical" soil methods have formed the basis for a sporadic progression of wet-chemical techniques during the last 15 years, to better characterize heavy metals in a variety of environmental materials, including marine sediments. The trend has been an ongoing increase in the number and kinds of treatments used in extraction schemes (Table 4). The most advanced procedures attempt to sequentially release metals from specific, operationally-defined sediment phases (e.g., Table 5), as reviewed by Forstner & Wittmann (1981, pp. 238-244).

It was decided that sediments from all 39 stations sampled in this study, would be analyzed for both total, and extractable (single, cold leach: Chester & Hughes, 1967) metal concentrations. These are common strategies world-wide, facilitating a comparison with data from other regions. The eight-step sequential extraction scheme (SES) was included in this study (Table 5), to provide a more detailed description of the geochemical partitioning

TABLE 4

PRINCIPAL WET-CHEMICAL METHODS
TO INVESTIGATE HEAVY METALS
IN ENVIRONMENTAL SOLIDS

Method of Investigation	Example References
Single, COLD LEACH with Dilute Acid	Chester & Hughes, 1967 Piper, 1971 Duinker et al., 1974 Chester & Voutsinou, 1981 Piotrowicz et al., 1981 Voutsinou-Taliadouri, 1981
Single, HOT ATTACK with Concentrated Acid	Carmody et al., 1973 Anderson, 1974 Taylor, 1974 Eisler et al., 1977 Sinex et al., 1980
SHORT SEQUENCE: includes 2 or 3 partial extractions	Presley et al., 1972 Grieve & Fletcher, 1976 Gibbs, 1977 Eisenreich et al., 1980 Kitano et al., 1981 Ellaway et al., 1982 Badri & Aston, 1983
LONG SEQUENCE: includes 4 to 6 partial extractions	Gupta & Chen, 1975 Engler et al., 1977 Hoffman & Fletcher, 1979 Tessier et al., 1979 Kitano & Fujiyoshi, 1980 Zielinski et al., 1983 Calmano & Forstner, 1983
TOTAL DECOMPOSITION	Medlin et al., 1969 Buckley & Cranston, 1971 Bruland et al., 1974 Eggimann & Betzer, 1976 La Brecque, 1979

of the metals than is possible with the single-step procedure of Chester & Hughes (1967). The multi-step procedure, with its superior discrimination and scope, can also aid in the identification of the metal fractions attacked by the popular Chester & Hughes (1967) reagent.

(b) precautions to minimize heavy-metal contamination

In all the following procedures, double or triple distilled-deionized waters were used in the reagents, rinsing solutions, and extract dilutions, to minimize heavy-metal contamination. All glassware, polyethyleneware, and utensils were routinely acid-rinsed to remove metal contaminants on their surfaces. Before all extracts were filtered, the filter paper (Millipore Type HA, 0.45 μm pore size) and the associated glassware, were washed with 5% HCl, and then rinsed with distilled water. All final solutions from the sediment extracts and digests were stored in acid-washed polyethylene bottles with polyseal caps, to prevent any contamination during storage and minimize volume reduction due to evaporation. For every type of digestion or extraction, two or more reagent blanks were taken through every aspect of the procedures, to correct for all systematic sources of contamination.

(c) release of the entire contents of metals from the sediments

The total abundances of V, Cr, Mn, Fe, Co, Ni, Cu, and Zn in the 39 sediments, were determined from 0.5 gm quantities of the fine powders (Figure 9), following the method outlined by Rader and Grimaldi (1961, p. A32). Briefly, the samples were placed in platinum crucibles and heated slowly to ash the organic matter (to render it soluble in the digesting solution). The ashed samples were next transferred into teflon dishes, and digested to dryness on a hot plate with 20 ml each of 48% hydrofluoric and 70% nitric acids (HFNA). The dry residues were digested a second time to dryness with 20 ml of the 70% nitric acid, to obtain the metals in solution as nitrates. The final residues were dissolved in 25 ml of 10% nitric acid, and then stored in the cleaned polyseal bottles.

(d) release of the extractable metal fractions

To mobilize the extractable (single, cold leach) heavy-metal fractions, one-gram portions of the lightly-disaggregated separates (Figure 9), were added to 100 ml polybottles with 50-ml solutions of 1-M hydroxylamine hydrochloride in 25% acetic acid (HHAA), and mechanically agitated for four hours (Chester & Hughes, 1967). The

resulting extracts were centrifuged, suction filtered, evaporated down, made up to 25-ml volumes with distilled water, and also stored in cleaned polyseal bottles.

Sequential Extraction Scheme

(a) rationale of the adopted scheme

In attempting to detail the chemical associations of the metals in Simpson Lagoon sediments, two to six replicate portions from six stations (2, 13, 18, 23, 28, and UG-1), which represent a range of grain-size, clay mineralogy, and carbonate and organic-carbon contents (Appendix II), were subjected to a sequential extraction scheme (SES) containing eight different chemical treatments (Table 5). This SES is one of the most elaborate strategies developed to date to elucidate the partitioning of metals in sediments.

Unlike many extract designs in use today (Table 4), the eight-step procedure adopted herein (Table 5) includes a treatment (Step 2: cation exchange resin) to selectively dissolve carbonate phases (Forstner & Patchineelam, 1980; Tessier et al., 1980; Forstner et al., 1981; Forstner & Wittmann, 1981; Hong & Forstner, 1983; Meguelatti et al., 1983; Rapin, 1983; Rapin & Forstner, 1983). The present scheme also, with Step 5 (oxalate) and

TABLE 5

SEQUENTIAL EXTRACTION PROCEDURE OF THIS STUDY, OUTLINING THE CHEMICAL TREATMENTS AND THEIR CORRESPONDING TARGETS TO BE MOBILIZED FROM THE SEDIMENT

Step	Chemical Treatment	Target Mobilized	Reference
1	0.2 N $MgCl_2$-triethanolamine; pH 8; 5 minutes	Exchangeable phase*	Jackson, 1958, p. 88
2	Cation exchange resin** (8 gm) in nylon net bag; pH 5; 8 hours; 200 ml	Carbonates	Sibbesen, 1977; Deurer et al., 1978
3	Acetylacetone in benzene (5:95); 250 hours	Organic-complexed metals	Giovannini & Sequi, 1976
4	0.1 M hydroxylamine-HCl in 0.01 M HNO_3 (pH 2); 30 minutes; continuous agitation	Mn and Easily-reducible oxides	Chao, 1972
5	0.2 M acid ammonium oxalate; pH 3.3 buffered; 2 hours; continuous shaking	Amorphous Fe oxides, some Organic matter	Daly & Binnie, 1974
6	4% Na dithionite in 0.2 M citrate buffer (pH 4.8); 30 minutes; 50° C	Crystalline Fe and moderately-reducible oxides	Coffin, 1963
7	2% $KClO_3$ in 0.5 N HCl; pH 0.3; 24 hours	Fe and Trace-metal sulfides from the dithionite treatment	Olade & Fletcher, 1974; Agemian & Chau, 1977
8	Digest to dryness with 48% HF & 70% HNO_3; 1:1 volume ratio	Residual, resistant minerals	Rader & Grimaldi, 1961, p. A32

* Includes interstitial water and water-soluble fractions.
** Dowex 50W-X8, 20-50 mesh, meq/g = 5.09 (dry wt basis).

Step 6 (dithionite), attempts to differentiate between amorphous and crystalline iron oxides respectively (Hoffman & Fletcher, 1979; Forstner & Wittmann, 1981, p. 243, Table 70; Learned et al., 1981; Zielinski et al., 1983; Chao, 1984). Another attractive feature of the design outlined in Table 5, is a treatment with an oxidizing agent under acidic conditions (Step 7: $KClO_3$ in dilute HCl). This is to recover any metal sulfides which may have precipitated (preventing their filtration) during the disproportionation of the dithionite reagent in Step 6 (Hoffman & Fletcher, 1979; Learned et al., 1981). Forstner & Wittmann (1981, p. 239) have listed the major chemical phases of sediments, and the corresponding treatments presently in use to selectively mobilize them. Details of the SES followed in this study are outlined in Table 5 and described in the following subsection (b).

(b) operation of the extraction sequence

Two-gram portions of the lightly-disaggregated separates (subsamples) were used for the sequential-extraction experiments (Figure 9). Except where noted otherwise, all extractions were carried out at room temperature, in 50 ml of extraction solution, and with occasional agitation. In order to differentiate the operationally-defined chemical phases (Table 5), the sub-

sample portions were sequentially subjected to the conditions of each corresponding treatment, starting with Step 1. The extracts obtained from each treatment were centrifuged, and then suction filtered using a Millipore glass-filtering apparatus. The sediment residues were then washed first with 30 ml of 0.1 N $MgCl_2$, followed by 30 ml of distilled water; the washings were filtered and thus added to the extracts. The residues were washed in this way to remobilize (mass action of Mg^{++}) any liberated metals which may have re-adsorbed to the sediment particles before the extracts were filtered. The washed residues were then subjected to the next treatment in the eight-step sequence. The filtered extracts from each step, combined with their associated washings, were evaporated down and made up to volume with distilled water according to their cited procedures (Table 5), and then stored in cleaned polyseal bottles.

(c) novel aspects of the adopted scheme

To extract metals associated with organic matter, a complexing agent in a non-polar solvent: acetylacetone in benzene (Step 3 in Table 5), was selected instead of one of the more commonly used oxidizing agents (e.g., hydrogen peroxide). Although the technique chosen here may be less quantitative, it is believed to be more specific

for organic-bound metals than the extremely disruptive conditions imposed by oxidizing agents (Stevenson, 1982, pp. 36-42). It is also difficult to obtain metal-free hydrogen peroxide.

It is sometimes necessary to slightly modify cited procedures in response to many criteria, including the nature of the material being studied, or the need for less time-consuming operations. In this study, barium chloride ($BaCl_2$) was replaced by $MgCl_2$ in Step 1 (Table 5) to avoid formation of $BaSO_4$ precipitates, which result because of the abundant sulfate in marine sediments. This was to preclude the possibility of any metals co-precipitating with the sulfate. Step 2 combines the nylon bag technique of Sibbesen (1977), with the use of an exchange resin adopted by Deurer et al. (1978). The nylon bag allows a convenient separation of the exchange resin from the sediment particles after each treatment. Additionally, to simplify the oxalate procedure in Step 5 (Table 5), a single, two-hour reaction was followed instead of repeating a one-hour reaction as in Daly & Binnie (1974). It has been shown that the oxalate reagent increasingly extracts Fe with time, and the choice of reaction time to differentiate amorphous Fe oxides is somewhat arbitrary (Schwert[illegible]1, 1973).

Atomic Absorption Analysis of the Liberated Metals

(a) instrument calibration

The concentrations of V, Cr, Mn, Fe, Co, Ni, Cu, and Zn in the solutions made from the total digests and partial extracts, were quantified using atomic absorption spectrophotometry (AAS). The instruments (following subsection) were calibrated each time with standards (known metal concentrations) similar in composition to the appropriate sample solutions (digest or extract). This was to eliminate errors in measurements due to different absorption interferences for the standard and sample solutions. A modified version (much faster) of the method of standard additions (Perkin-Elmer, 1982, p. 9.4), was employed for this purpose.

The modification consists of contributing small aliquots from each sample solution into one blended stock. Relatively small volumes of combined AA standards (Fisher Scientific company) were added to undiluted portions of this stock, to obtain mixtures with ionic concentrations nearly matching the average of the sample solutions. Collective standard additions were produced in this way for every type of digest or extract. It is believed this compromise of the standard-additions technique is justified because the lithology, and therefore the major-element

composition, of all the sediments within the limited extent of western Simpson Lagoon is probably quite uniform (cf. clay minerals in Appendix II-C). Additionally, the ranges in the 39 stations for concentrations of all the metals is less than a factor of three (Appendices II-D,E). Standards matching the average of their corresponding sample solutions should therefore have absorption interferences similar to each individual sample solution.

(b) spectrophotometers used

All metal analyses were accomplished using Perkin-Elmer spectrophotometers equipped with Deuterium Arc background correctors. The faster and more precise method of flame atomic absorption (603 unit) was employed whenever possible. The flameless sampling accessory, HGA graphite furnace, was used only if the metal concentration was below the sensitivity of the flame technique.

The HF-HNO_3 digests (HFNA) were analyzed for all eight metals on a Model 603 unit (flame). Analyses were performed for each metal under the standard operating conditions of the instrument (Perkin-Elmer, 1982). For the extractable fractions (HHAA; Chester and Hughes, 1967), V, Cr, Co and Ni were run on a Model 360 unit equipped with an HGA-2100 graphite furnace. The other metals, which had relatively higher solution concentrations, or

greater analytical sensitivities, were analyzed with the 603 unit.

For most of the sequential extracts, Ni, Co, Cr, and V were analyzed on a subsequently available Model 5000 unit equipped with an HGA-500 graphite furnace (Appendix VIII-H). This furnace allows the operator to program up to nine steps of temperature ramping, a feature which improves the sensitivity and precision for analysis of complex solutions. For Cu, extracts from Steps 1-3 (Table 5) were analyzed with the HGA-500 graphite furnace, the remaining steps with the 603 flame unit. The concentrations of Fe, Mn, and Zn in all sequential extracts were quantified with the 603 unit. All metals were sufficiently concentrated in Step 8 digests to be analyzed with the Model 603 unit (Appendix VIII-H). Analyses on the graphite furnaces were also performed under standard operating conditions (Perkin-Elmer, 1977).

Statistical Methods

(a) the importance of reporting the precision and accuracy of measurements

Due to the dynamic physical and chemical processes in nearshore ecosystems, coastal sediments are very complex and highly variable materials. Lithological and

chemical parameters in nearshore sediments often exhibit considerable local variations (within meters); subsamples separated in the laboratory may also show significant differences. Of relevance to partitioning studies, organic and inorganic colloidal phases may be in such close association that their discrimination is very difficult by present methods. Additionally, Fe-oxides may occur as amorphous precipitates, aged crystalline minerals, or as intermediate subcrystalline varieties. The acknowledgement of these complexities implies that knowledge of the environmental and analytical variabilities, and analytical accuracy, is critical to the evaluation of constituents of nearshore sediments.

(b) estimation of environmental and analytical variations in the sediment parameters

The variability in measurements of sediment parameters can be delineated into three main groups: regional diversity, sample heterogeneity, and analytical precision. In this study, regional diversity was evaluated in terms of inter- and intra-station variations, sample heterogeneity in terms of intra-sample and intra-subsample variations, and analytical precision in terms of instrument calibration and operation, and operator calculations of the final values. Figures 10 through 16

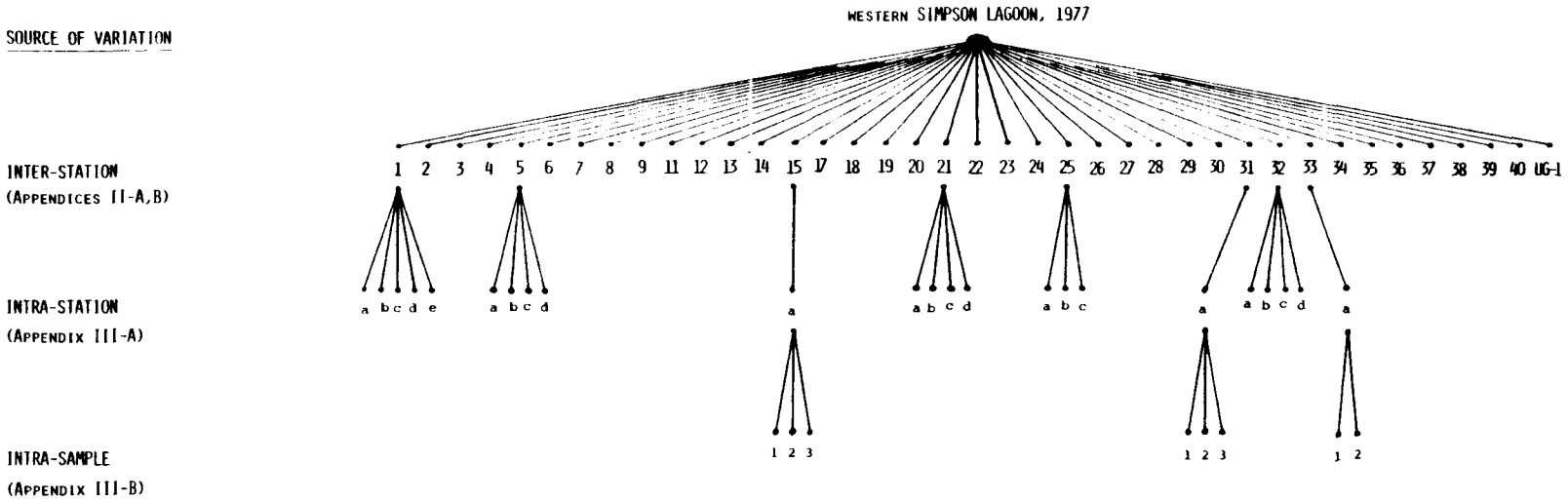

Figure 10. Replicates taken to compare sources of variation in estimates of grain-size parameters.

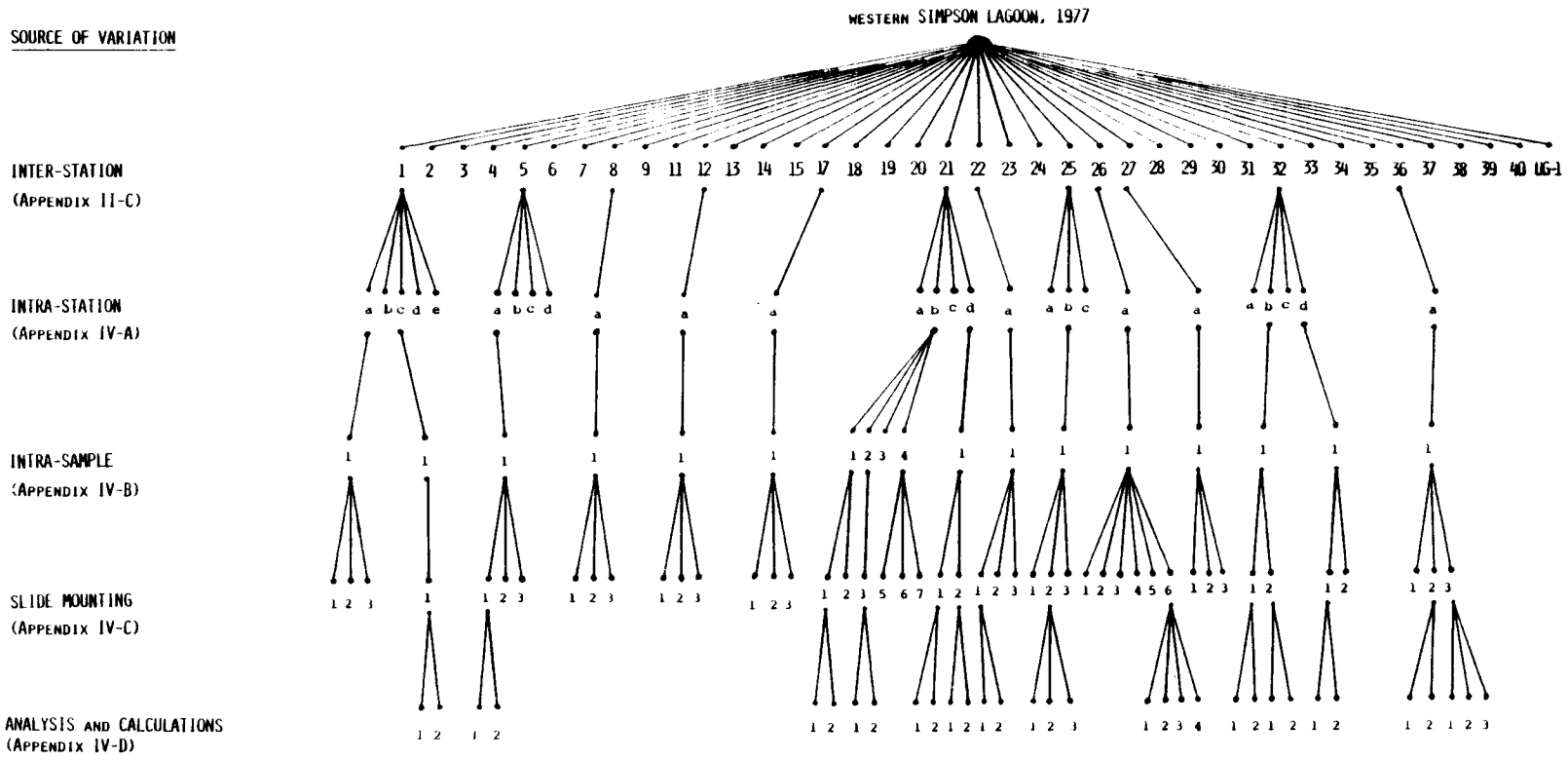

Figure 11. Replicates taken to compare sources of variation in estimates of clay-mineral proportions.

depict which of these sources of variation were examined in this study for each class of variables (e.g., clay minerals in Figure 11). The magnitudes of the variations explained by each source (except inter-station), were obtained by averaging separate variance calculations on several samples, each with corresponding replicate data (Appendices III through VIII). This technique of pooling sample variances is described in many general statistical texts; the variance estimates produced in this way are listed in Appendix X, as standard deviations (s).

(c) accuracy of the analytical methods

The knowledge of the closeness of the value reported for a variable, to its true abundance in the sediment, is possibly even more critical to evaluating the environmental significance of that variable, than is documenting the dispersion of the reported value. The accuracies of the measurements of total carbon and carbonate were estimated by the respective analyses of pure dextrose and calcium-carbonate standards interspersed between samples (Appendices V-C,E). The accuracies of the estimates for the total-metal (HFNA) concentrations were checked by comparing total abundances determined in this study for USGS AGV-1 and BCR-1 Standard rock powders, with results published by Flanagan (1969, 1973).

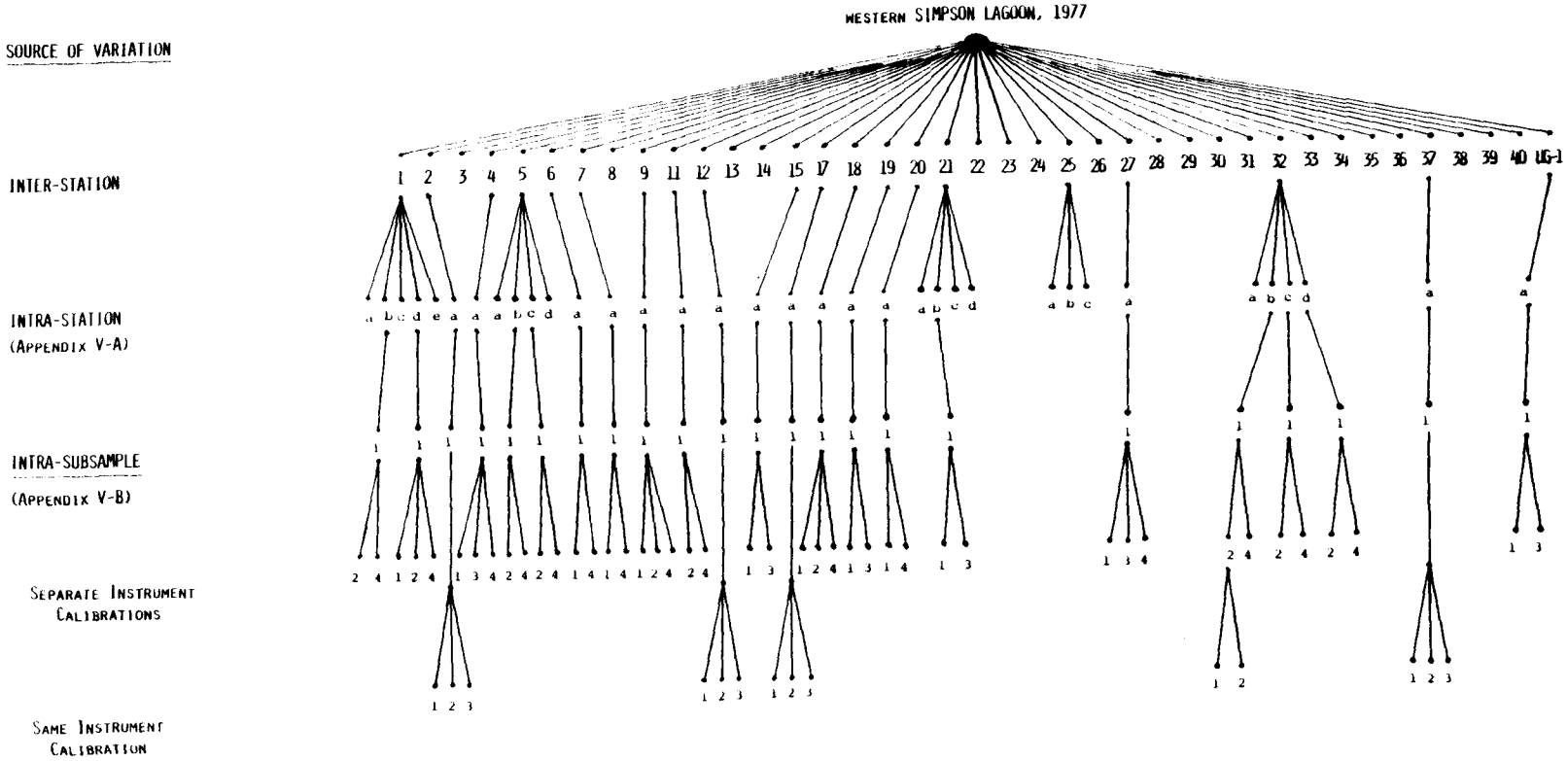

Figure 12. Replicates taken to compare sources of variation in measurements of total-carbon contents.

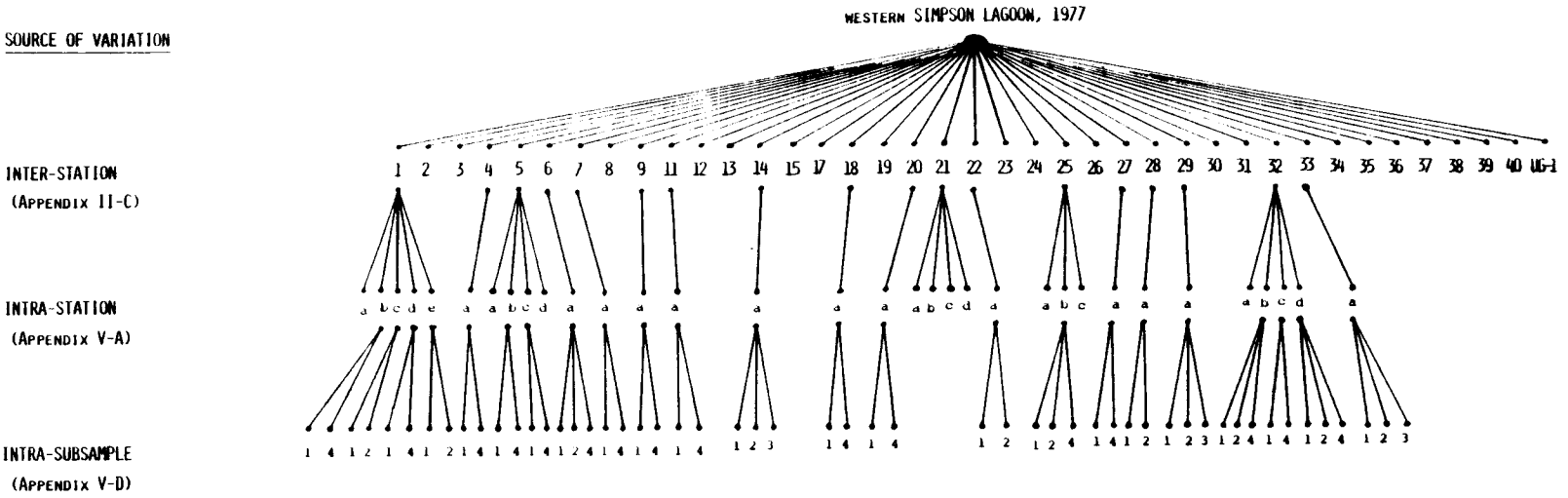

Figure 13. Replicates taken to compare sources of variation in the determinations of carbonate contents.

The accuracies of the total-metal concentrations calculated from the eight-step SES were assessed in the following way. The amount of each metal mobilized in each of the sequential treatments was added together. These calculated totals were then compared to the results obtained from a single, $HF-HNO_3$ digestion of a separate 0.5-gm portion (Appendices II-D,E). Comparisons were accomplished for subsample portions from two stations (2 & 23), with the results presented in Appendix XI.

(d) multivariate statistics

In attempting to determine the major factors controlling heavy-metal distributions in the sediments of Simpson Lagoon, factor, cluster, and stepwise discriminant analyses were performed on a raw data matrix consisting of 28 of the variables measured (e.g., Figure 17) for the 39 stations sampled (Figure 8, Appendix I). Before these analyses, however, the concentrations of gravel, extractable (HHAA) Mn, and organic carbon were first transformed to their natural log values, because these variables had severe skewed-frequency distributions.

The Biomedical Computer Program BMDP-4M (Dixon, 1981), was employed for the R-mode (variables) factor analysis. Initial (unrotated) factor extraction was by principal components (Dixon, 1981, p. 486), which was fol-

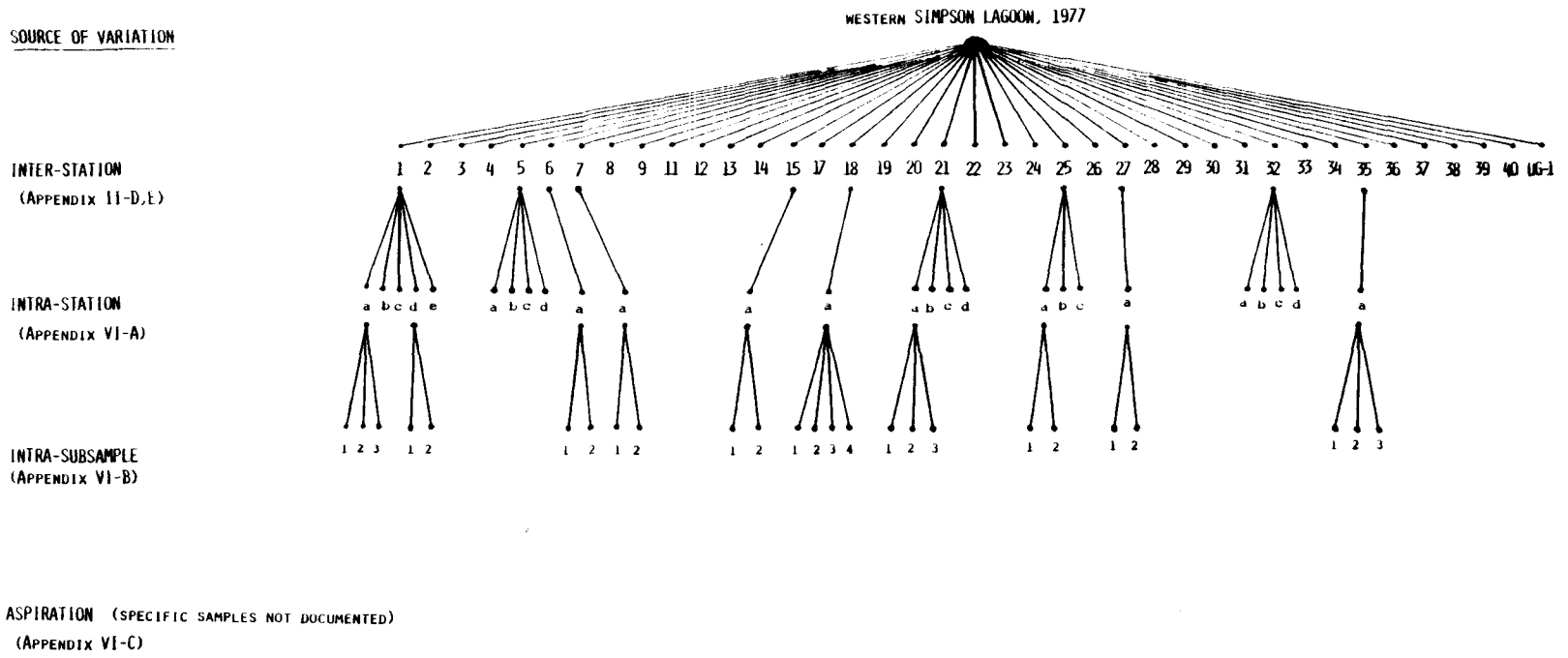

Figure 14. Replicates taken to compare sources of variation in estimates of total-metal concentrations.

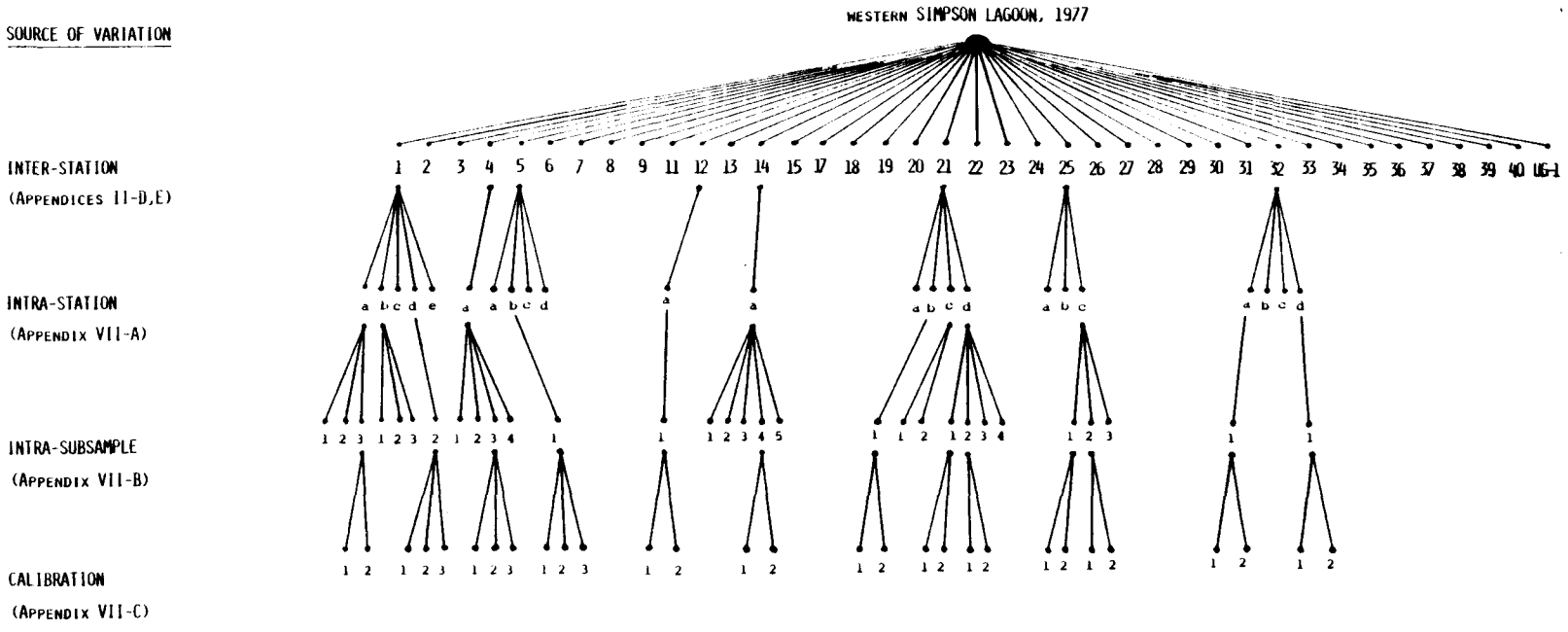

Figure 15. Replicates taken to compare sources of variation in determining the extractable (HHAA) fractions of metals.

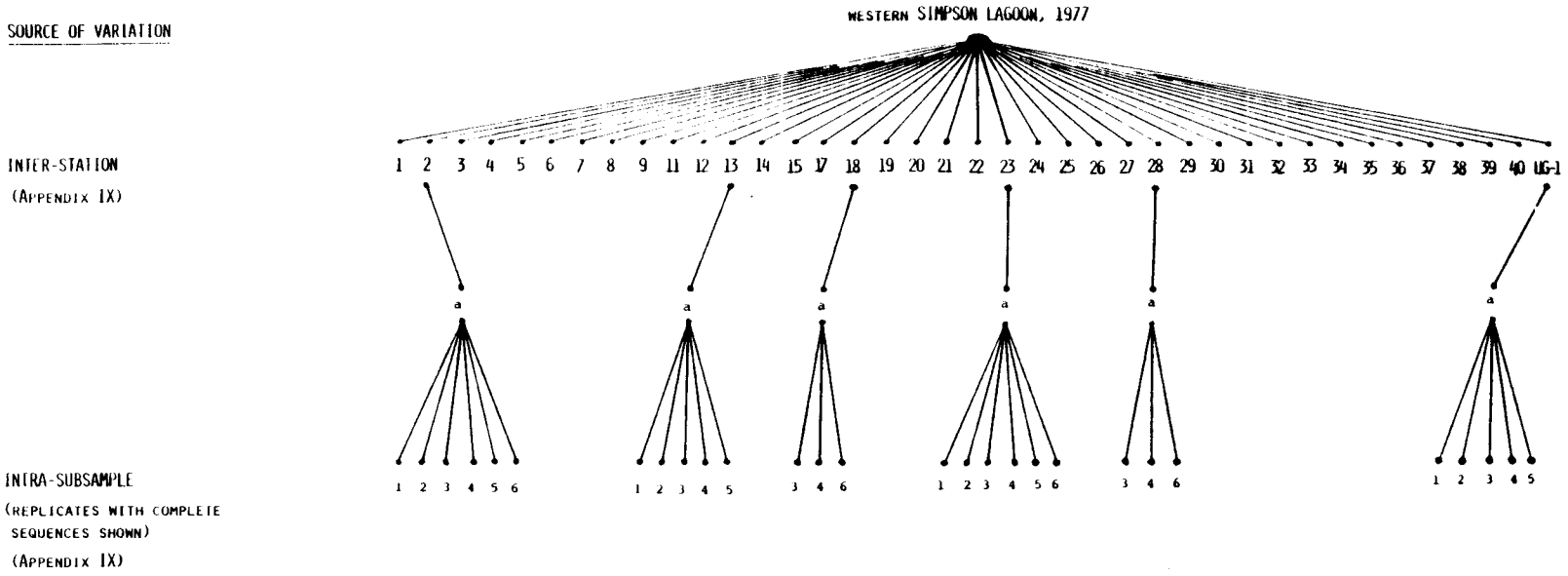

Figure 16. Replicates taken for comparison of the inter-station and intra-subsample sources of variation in metal fractions elucidated by the eight-step sequential extraction scheme (SES).

lowed by a Varimax factor rotation. Cluster analysis was performed with the BMDP-2M program (Dixon, 1981, pp. 456-463), with station clusters joined using the centroid linkage algorithm. The BMDP-7M program (Dixon, 1981, pp. 519-537), was used for the stepwise discriminant analysis. Discrimination was performed on the three main station groupings obtained from the cluster analysis (Figure 18).

(e) analysis of variance on heavy-metal fractions

The reproducibility of the sequential extraction scheme (SES) was in doubt, due to its great complexity, and numerous requirements for handling the samples. To examine the repeatability of its results, the SES was performed on replicate subsample portions, taken from each of the six stations chosen for the partitioning study (Figure 16, Appendix IX). A one-way analysis of variance (ANOVA) was then undertaken, to compare the inter-station and intra-subsample variances of the metals extracted by the eight-part scheme (Table 5). The details of the ANOVA are described in detail in Appendix XIII.

RESULTS

Precision of Analyses

(a) non-metallic parameters

The summary of replicate analyses for selected grain-size parameters and clay minerals (Appendix X-A), reveals that the major source of variation in their measurement is at the inter-station level. This relationship is particularly dramatic for the grain-size parameters. The smaller contrast between the sources of variation in the clay-mineral estimates, may possibly be due to a relatively more uniform distribution of clay-size particles (<2μm) in the sediments throughout Simpson Lagoon. For the concentrations of carbonate and organic carbon in the sediments of Simpson Lagoon, between-station variations are also significantly (> 3 times) greater than variations within stations. It should be noted also that greater precision for the analysis of subsamples appears to be achieved when running replicates during the same calibration of the instrument (Appendix X-B).

(b) precision of analyses of total and extractable metals

Inter-station variances for total and extractable (HHAA) metal abundances (for all the metals), are much greater than all the other sources of variation combined (Appendices X-C,D). Clearly, an F-test is not required in order to conclude that stations in Simpson Lagoon can be discriminated on the basis of these heavy-metal fractions.

An additional source of variation was identified and estimated for extractable metals. It seems that for most of the metals, approximately twice as much variation occurs between repeated aspirations of the same sediment extract, if the two measurements are made from separate instrument calibrations (Appendices VII-C, X-D).

Data summarized in Appendices X-A to X-D reveal that, in general, the standard deviations (s) for intra-station sources of variation are < 10%. Exceptions are, expandable minerals: 25%; carbonate: 12%; organic carbon: 20%; total Co: 14%; total Cu: 20%; and extractable (HHAA) Cr, Mn, Fe, Co, Ni and Zn: with 20%, 23%, 14%, 15%, 20% and 12%, respectively.

(c) reproducibility of results for the sequential extraction procedure (SES)

The statistics presented in Appendices X-E to X-I show large variabilities (s) in the results obtained for the first five steps of the extraction sequence. Deviations between replicates for amounts of heavy metals extracted are generally between 50% to over 100% for some treatments. The acetylacetone in benzene (Step 3), and the acidified hydroxylamine hydrochloride (Step 4) reagents suffered the poorest reproducibilities (Appendices X-G,H). The very small amounts (sometimes ng/g) of metals mobilized by some of these treatments is the likely reason for the large errors. Minor deviations in the extraction procedures, and errors due to instrument drift are relatively more significant when analyte concentrations are small.

In order to obtain more precise, although operationally-defined extraction data, and considering the selectivity of the treatments after Step 4 for Fe phases (Appendix VIII), the data from the first seven treatments were combined into two descriptive groups. Steps 1-4 (Table 5) became represented as Group I, designated "Leachable Fractions", and Steps 5-7 became represented as Group II, designated "Fe Phases". This grouped data

is listed in Appendix IX. Inter-station and intra-subsample (pooled) standard deviations for the summed data represented by Groups I and II are presented in Appendices X-L to X-N.

A comparison of the intra-subsample variances for each kind of heavy-metal data produced in this study was next made. This includes the sequential extractions (Table 5), the combined extraction Groups I and II (Appendix IX), the single extracts (HHAA), and the total digests (HFNA). The comparison was accomplished by calculating their corresponding coefficients of variation (cv) from the statistical parameters of Appendix X. The results are listed in Tables 6 and 7, along with the mean values of the replicates for each treatment or group.

A marked improvement in reproducibility was obtained with the reduction from seven steps to two extraction groups. The two-group representation of the sequential extraction data obtained in this study has the added advantage in differentiating the more easily mobilized metal phases (Group I: exchangeable, carbonate, organic complexes, Mn oxides), from the more resistant Fe phases (Group II: amorphous to crystalline Fe oxides).

TABLE 6

MEAN CONCENTRATIONS (μg/g) AND COEFFICIENTS OF VARIATION (cv) FOR THE AMOUNTS OF V, Cr,[1] Mn & Fe IN OPERATIONALLY-DEFINED FRACTIONS OF SEDIMENTS IN SIMPSON LAGOON

Defined Fraction	VANADIUM $\bar{X}$	cv	CHROMIUM $\bar{X}$	cv	MANGANESE $\bar{X}$	cv	IRON $\bar{X}$	cv
BY TREATMENT[2]								
Step 1	0.25	72	0.07	135	21	23	15	69
Step 2	0.88	65	0.39	80	48	46	177	55
Step 3	0.44	102	0.07	112	17	155	478	84
Step 4	0.61	115	0.06	140	20	87	151	87
Step 5	3.5	34	0.74	53	14	94	3290	27
Step 6	2.9	17	0.83	90	28	12	3560	9
Step 7	2.2	30	2.1	21	19	35	1980	24
BY SEQUENCE[3]								
Group I	2.2	32	0.71	44	96	20	668	48
Group II	8.8	16	4.0	22	59	17	8740	12
I + II	11.0	17	4.8	21	158	15	9290	8
HHAA[4] EXTRACTS	4.3	8	1.2	12	141	7	2340	9
HFNA[5] DIGESTS	70	4	43	5	200	2	18400	2

[1] Based on the intra-subsample data from Appendix X.
[2] Replicate data in Appendix VIII.
[3] Replicate data in Appendix IX.
[4] Replicate data for hydroxylamine hydrochloride-acetic acid extracts (HHAA) are found in Appendix VII.
[5] Replicate data for hydrofluoric-nitric acid digests (HFNA) are found in Appendix VI.

TABLE 7

MEAN CONCENTRATIONS (μg/g) AND COEFFICIENTS OF VARIATION (cv) FOR THE AMOUNTS OF Co, Ni,[1] Cu & Zn IN OPERATIONALLY-DEFINED FRACTIONS OF SEDIMENTS IN SIMPSON LAGOON

Defined Fraction	COBALT $\bar{X}$	cv	NICKEL $\bar{X}$	cv	COPPER $\bar{X}$	cv	ZINC $\bar{X}$	cv
BY TREATMENT[2]								
Step 1	0.04	44	0.18	19	0.55	31		
Step 2	0.42	64	2.2	64	1.8	89	8.5	54
Step 3	0.18	110	0.04	146	2.9	76		
Step 4	0.21	81	0.41	107	0.20	110	2.2	59
Step 5	0.59	83	1.8	56	3.6	50	5.5	109
Step 6	1.55	21	2.4	31	1.0	42		
Step 7	0.85	53	3.3	19	2.6	46		
BY SEQUENCE[3]								
Group I	0.87	36	3.0	57	4.8	50		
Group II	2.9	20	7.3	8	7.4	32		
I + II	3.6	19	9.6	17	11.7	25		
HHAA[4] EXTRACTS	2.0	9	3.1	14	3.0	17	12.3	6
HFNA[5] DIGESTS	7.5	7	22	4	15	16	70	6

1 Based on the intra-subsample data from Appendix X.
2 Replicate data in Appendix VIII.
3 Replicate data in Appendix IX.
4 Replicate data for hydroxylamine hydrochloride-acetic acid extracts (HHAA) are found in Appendix VII.
5 Replicate data for hydrofluoric-nitric acid digests (HFNA) are found in Appendix VI.

Accuracies of the Analytical Methods

(a) total carbon, carbonate, and total metals

Replicate analyses of dextrose standards for total carbon suggest on average, a 4% underestimation (96% efficiency) in this study for total-carbon contents of Simpson Lagoon sediments (Appendix V-C).

Replicate runs on a calcium carbonate standard during the gasometric analyses suggest a ±7% possible error for estimates of the magnitudes of the carbonate contents of the sediment samples (Appendix V-E).

The estimates for the total concentrations of the metals are considered to have good accuracy, based on results shown in Appendix VI-D. Results from digests performed on standard rock powders in this study agree closely with average values reported by Flanagan (1969, 1973), which were based on voluminous multi-laboratory contributions. Nickel, however, is the exception: results on the BCR-1 rock standard in this study are about 30% of the reported average (Appendix VI-D). Total nickel concentrations reported herein should therefore be accepted with caution.

(b) accuracies for the results of the sequential extraction experiments

To examine the accuracy of the SES, the sum of the eight, partial extracts were compared to results of analysis on a single, total digest (HFNA), for samples from two stations (Appendix XI). It was not possible to obtain data on V and Co for Step 8 digests, due to exhaustion of the solution sample before their analyses. Generally, for each metal the sum of the eight steps is similar or lower than results from a single, HFNA digestion on a separate, whole, subsample portion. Nickel for SL877-2, however, is especially underestimated by the eight-step sequence, compared to a single digest (Appendix XI). In contrast, instances where the eight-step sum exceeds the values reported in Appendix II are Zn for station 2, and Cr and Fe for station 23 (Appendix XI).

Intra-subsample standard deviations (s) from Appendix X-C were included in Appendix XI. The difference between the eight-step sum and single-digest values for most metals is much greater than this "s" (Appendix XI), suggesting that subsample variance is not a major factor in the discrepancies between the two estimates of total heavy-metal concentrations. The SES and whole-digest estimates of the gross metal concentrations are also listed in Appendices XII-A and XII-D.

Results of the Multivariate Statistical Analyses

(a) factor analysis

Results of R-mode factor analysis (variables) are shown in Figure 17. The number of factors obtained was limited to those with eigenvalues (amount of variance explained) greater than 1.0 (Dixon, 1981, p. 482). The resulting five factors account for 88% of the total variance in the data matrix generated for Simpson Lagoon (Figure 17). Communalities of each variable were obtained from the five factors after one iteration (Dixon, 1981, p. 482), and provide an indication of the proportion of the variablility associated with any one variable that is explained by the five-factor model (Tripathi, 1979). The factor analysis was performed on a data matrix consisting of 28 textural, clay mineralogical, and chemical variables of the sediments (Figure 17, Table 8), versus 39 cases representing the sampling stations (Appendix II).

Factor 1, which has immediate relevance to the present geochemical study, accounts for 56% of the total variance in the data set. Clay and silt content, mean grain size, and all metals except Mn, have > 0.75 loadings onto Factor 1 (Figure 17). This suggests an enrichment of metals in the fine fraction of the sediments. Carbonate and organic carbon show significant but slightly

Variable	FACTOR 1	2	3	4	5	Commu-nality
(HFNA)Cr	0.94					0.97
(HFNA)Ni	0.93					0.97
(HHAA)V	0.92					0.90
Clay	0.91					0.87
(HHAA)Zn	0.90				-0.26	0.92
(HFNA)V	0.89	0.33				0.92
(HFNA)Zn	0.89	0.29				0.91
(HFNA)Co	0.89					0.85
(HFNA)Fe	0.88	0.38				0.96
Mean Size	0.87	0.33				0.94
Sand	-0.86	-0.34				0.92
(HFNA)Cu	0.86					0.75
(HHAA)Ni	0.84		0.33			0.89
(HHAA)Co	0.84		0.39			0.87
(HHAA)Fe	0.83		0.34			0.85
(HHAA)Cr	0.80		0.46			0.86
Silt	0.77	0.43	0.33			0.89
Carbonate	0.75	0.34	0.26			0.75
(HHAA)Cu	0.75	0.26			0.34	0.81
(HFNA)Mn	0.71		0.59			0.90
Organic Carbon	0.65			0.29	-0.50	0.83
Depth	0.28	0.83				0.85
Expandable Minerals		0.81		0.26	-0.25	0.84
Gravel		-0.39	-0.69	-0.26	0.38	0.85
(HHAA)Mn	0.65		0.66			0.90
Chlorite				0.87		0.79
Illite		-0.66		-0.68		0.95
Kaolinite				0.40	0.80	0.82
% VARIANCE EXPLAINED	55.9	11.2	8.6	6.4	5.4	

Figure 17. Sorted rotated factor loadings (pattern) for 28 variables. Loadings with absolute values < 0.25 have been omitted for clarity.

lower loadings onto Factor 1, suggesting a somewhat weaker correlation with grain size and metal contents.

(b) cluster analysis

Results for the cluster analysis, on the same 28-variable by 39-case data matrix as for the factor analysis, are shown in Figure 18 (grouping by station). At the amalgamated distance 4.497, three clusters stand out. Cluster 1 contains stations 1, 30, 2, 7, 34, and 20, all with sand content > 75% (Appendix II-A). Cluster 2 contains stations 15, 40, 24, 19, 25, 17, and 8, with sand contents between 60 and 75%. The remaining stations have < 60% sand contents and the majority are grouped in cluster 3. The exceptions are stations UG-1, 4, 18, 32, and 29, which are not clustered at the 4.497 distance (Figure 18).

The sediments taken from stations UG-1 and SL877-18 had unusually high organic matter, and were retrieved from coastal areas with high accumulations of detrital peat (Appendix I). In fact, at station 18, peaty mats overlied the sediment in a protected embayment, with reducing conditions in evidence, based on the observed black color of the surficial sediment, and odor of hydrogen sulfide (Appendix I). The anoxic environment at station 18 is unusual for Simpson Lagoon. As discussed under "Area of

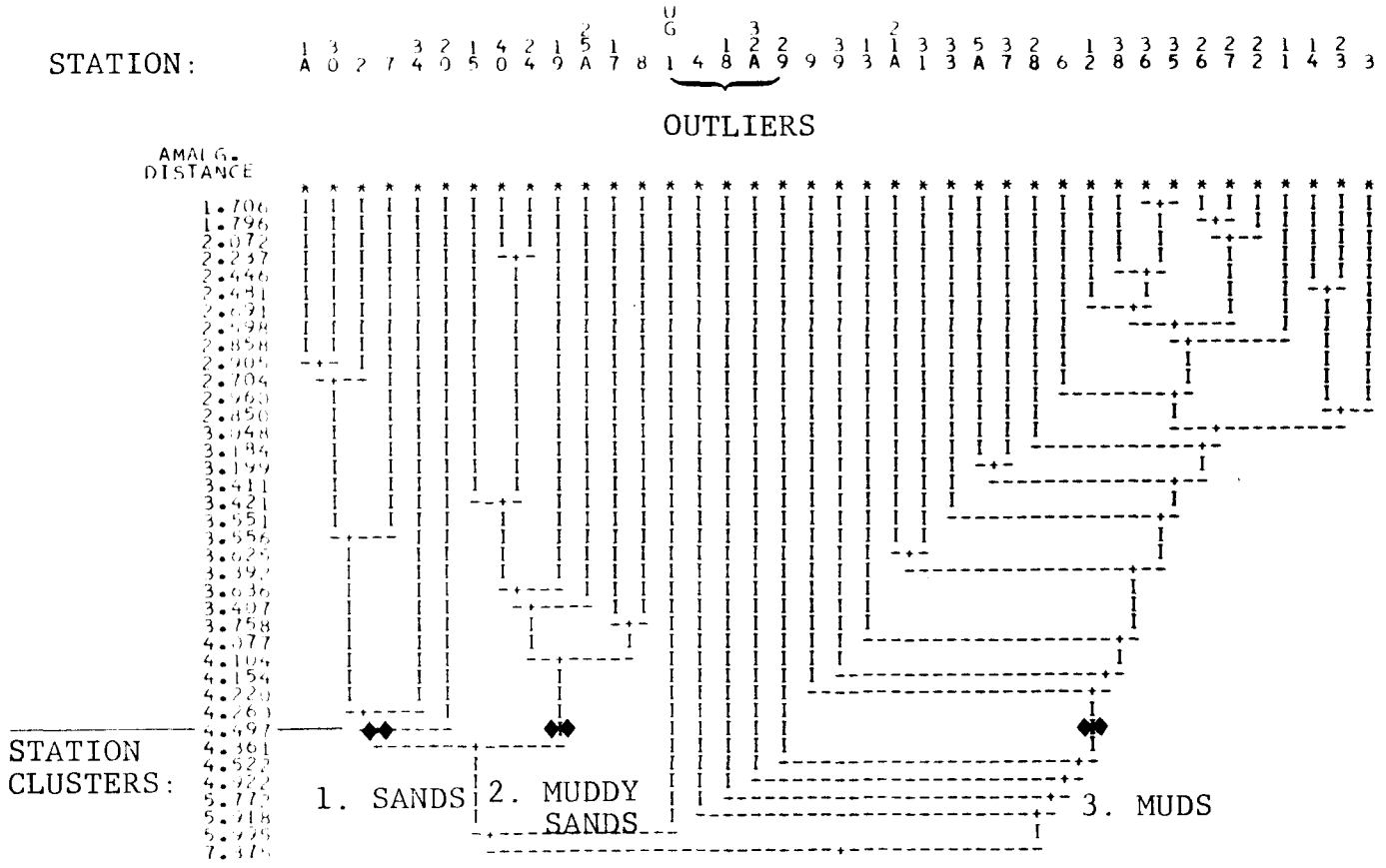

Figure 18. Vertical tree diagram of station clusters in western Simpson Lagoon, based on 28 variables. The three sediment classes shown were entered into the discriminant analysis.

Study" and observed in the sediment grabs (Appendix I), Simpson Lagoon sediments are considered to be well-aerated, with oxic conditions present in surface layers. In contrast, the sediment from station 4 was probably not clustered at the 4.497 distance because of its unusually high metal abundances (Appendix II-D).

The three clear clusterings in Figure 18 occur at a relatively large amalgamated distance (4.497). There are only a few close clusterings--small distance (e.g., stations 35,36). This may be due to a wide and continuous range of sediment texture across Simpson Lagoon. The large inter-station variances shown in Appendices X-A to X-D support this. It would seem that the systematic sampling in a grid pattern across the lagoon (Figure 8), assured the retrieval of sediments satisfactorily representing the range of sediment textures occurring in Simpson Lagoon.

(c) stepwise discriminant analysis

Results from the cluster analysis were applied to the discriminant analysis. Stations in clusters 1, 2, and 3 were first assigned to the respective classes: SANDS, MUDDY SANDS, and MUDS, based on their textural differences (preceding subsection (b), and Figure 18). The stations not clustered at the 4.497 level (UG-1, 4, 18, 32, and

29), were assigned to the OUTLIERS category. The OUTLIERS were not included in the calculations of the discriminant analysis, but would be assigned to one of the three textural classes by it.

Table 8 lists the means and standard deviations of the 28 variables for each of the three classes of sediments (same variables used in factor and cluster analyses). Influence of particle size is indicated, since the mean value for most of the variables is smallest for the SANDS, and largest for the MUDS (Table 8).

There are, however, three exceptions to this trend. The percentage of gravel-size particles in the sediments of Simpson Lagoon appear to increase according to another trend: MUDDY SANDS > SANDS > MUDS (Table 8). This may be related to the fact that for the MUDDY SANDS, stations 19 and 8 are located closer to shore (Figure 8) than the adjacent SAND stations of 20 and 7 respectively. In contrast, the distribution of illite follows an opposite trend to that of mud content. This seems to reflect well with the negative relationship between illite and depth in Factor 2 (Figure 17). It is possible that there is sorting of clay minerals based on grain size. Kaolinite covaries with gravel (Table 8), which presumably again suggests size sorting of this mineral.

TABLE 8

MEANS ($\bar{X}$) AND STANDARD DEVIATIONS (s) FOR 28 VARIABLES[1] IN THREE CLASSES OF SEDIMENTS FROM SIMPSON LAGOON[2]

Variable[3]	SANDS[4]		MUDDY SANDS[5]		MUDS[6]	
	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s
Depth (m)	1.6	0.60	1.6	1.0	2.3	0.38
Gravel*	2.8	1.8	4.6	0.85	1.7	1.3
Sand	84.5	9.3	64.9	2.6	26.5	11.8
Silt	8.2	6.9	18.7	4.6	55.6	11.8
Clay	3.9	1.6	10.3	1.8	17.3	3.7
Mean Grain Size (Ø)	2.2	0.63	3.3	0.35	5.4	0.49
Expandable Minerals	4.3	3.7	5.1	3.1	8.4	3.2
Illite	65.2	4.7	63.3	2.3	59.6	3.6
Kaolinite	11.0	1.4	11.4	0.98	10.9	1.1
Chlorite	19.5	2.3	20.1	0.69	21.1	1.4
Carbonate	3.3	1.6	4.9	1.6	9.6	1.1
Organic Carbon*	0.35	0.21	1.0	0.50	1.5	0.31
(HFNA)Fe	10.6	1.9	15.6	2.2	23.5	2.9
(HHAA)Fe	0.89	0.47	1.2	0.34	2.7	0.50
(HFNA)Mn	136	18	186	23	322	80
(HHAA)Mn*	4.1	0.27	4.5	0.32	5.1	0.40
(HFNA)Zn	33.5	4.7	60.7	14.1	87.2	7.5
(HHAA)Zn	3.9	1.3	8.8	2.5	15.9	2.5
(HFNA)V	38.5	6.2	58.6	4.7	82.8	9.3
(HHAA)V	1.7	0.53	2.8	0.55	4.8	0.84
(HFNA)Cr	22.0	3.4	35.3	2.6	55.7	6.7
(HHAA)Cr	0.26	0.22	0.64	0.42	1.4	0.34
(HFNA)Ni	11.1	1.5	18.4	1.5	27.9	2.4
(HHAA)Ni	0.78	0.30	2.8	1.4	4.2	0.67

TABLE 8

(Continued)

Variable[3]	SANDS[4] $\bar{X}$	SANDS[4] s	MUDDY SANDS[5] $\bar{X}$	MUDDY SANDS[5] s	MUDS[6] $\bar{X}$	MUDS[6] s
(HFNA)Cu	8.7	1.9	12.9	2.2	20.0	4.4
(HHAA)Cu	1.8	0.51	2.6	0.74	2.9	0.45
(HFNA)Co	4.2	0.85	6.6	1.1	8.6	0.89
(HHAA)Co	0.68	0.35	1.5	0.69	2.4	0.43

[1] The same variables included in the factor analysis (Figure 17).

[2] Based on cluster and discriminant analyses on samples from 34 stations of Simpson Lagoon.

[3] Except where noted, units are for grain-size parameters, $CO_3^=$ & OC: wt percent; for clay minerals: rel. percent; for Fe: 10^3 µg/g; for other metals: µg/g.

HFNA: Digested with $HF-HNO_3$.
HHAA: Extracted with hydroxylamine hydrochloride-acetic acid (Chester & Hughes, 1967).

[4] Represented by six SL877 stations: 1,30,2,7,34 & 20.

[5] Represented by seven SL877 stations: 15,40,24,19, 25,17 & 8.

[6] Represented by 21 SL877 stations: 9,39,13,21,31,33, 5,37,28,6,12,38,36, 35,26,27,22,11,14,23 & 3.

These station clusters are shown in Figure 18.

* Values listed are based on the natural logarithms of the original data.

Table 9 lists the classification functions (CF) computed in the discriminant analysis procedure. Variables used in computing these linear CF's were chosen in a stepwise manner. Variables which add the most to the separation of the sediment classes are input into the functions until no variable exceeds the F-to-enter level of 4.00 (Dixon, 1981, p. 536). The smaller set of predictor variables so obtained, discriminates between the criterion groups (classes) virtually as well as the entire set of variables (Kachigan, 1982, p. 228). The results of this stepwise procedure are that primarily Ni, and secondly gravel, are the two variables which discriminate the best between the three station classes delineated by the cluster analysis.

As shown in Table 9, there is a three-term function corresponding to each sediment class, consisting of a linear combination of values for gravel and (HFNA)Ni, each multiplied by a coefficient, plus an added constant. For example, the SANDS classification function is:

$$CF_{sands} = 0.91(Gravel) + 2.37[(HFNA)Ni] - 15.47$$

The values for the variables (Gravel) and [(HFNA)Ni] are in the respective units: weight percent and μg/g. Classification of a sediment sample is made by inserting its

gravel and (HFNA)Ni values separately into the three CF's (Table 9). Assignment of sediment class is made according to which CF has the highest computed score. All the stations in the OUTLIERS class were assigned to the MUDS class in this way.

TABLE 9

COEFFICIENTS AND CONSTANTS FOR FUNCTIONS[1] WHICH ASSIGN* A SAMPLE TO A SEDIMENT CLASS, BASED ON ITS GRAVEL AND (HFNA)Ni CONTENTS

Terms of the Classification Function	SEDIMENT CLASS		
	Sands	Muddy Sands	Muds
Coefficients			
Gravel#	0.91	1.40	-1.10
(HFNA)Ni	2.37	3.95	6.40
Constants	-15.47	-40.70	-89.58

[1] Computed by the discriminant analysis.
* Assigned to the sediment class having the highest score calculated from corresponding classification functions.
# Gravel = ln(Gravel + 0.1) + 3.

A symbol table for a scatter plot of canonical variables for Ni and gravel, and the plot itself, are shown in Figures 19a,b. The first canonical variable is the linear combination of the variables entered, gravel

and (HFNA)Ni, that best discriminates among the sediment classes. The second canonical variable is the next best linear combination orthogonal to the first one (Dixon, 1981, p. 523). The coefficients and constants of the canonical variables so derived are:

	Canonical Variables	
	one	two
Coefficients		
Gravel	0.30	-0.73
(HFNA)Ni	-0.48	-0.09
Constant	10.19	3.87

and the x & y coordinants for each case plotted in Figure 19b are calculated from these functions in a manner similar to the computations of the classification scores shown previously.

Sediment Class	Mean Coordinates x	y	Symbol For Cases	Symbol For Mean
Sands	5.78	0.81	A	1
Muddy Sands	2.79	-1.09	B	2
Muds	-2.58	0.13	C	3
Outliers	-1.97	0.08	D	4

Figure 19a. Mean coordinates and key to symbols used in scatter plot of canonical variables (Figure 19b).

The plot of canonical variables can be thought of as a two-dimensional projection of the discriminant scores,

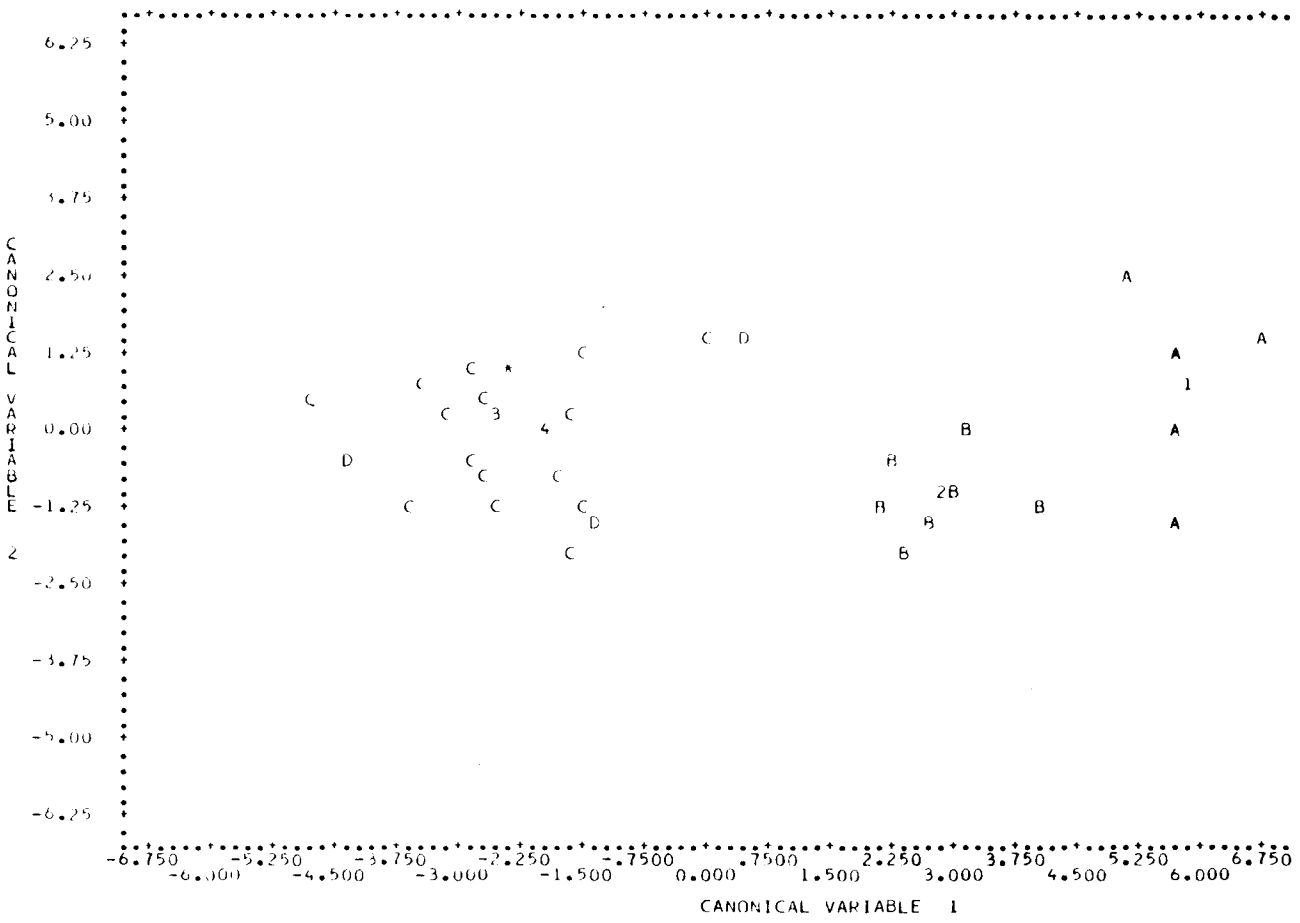

Figure 19b. Scatter plot for each case (station), of the coordinates for the canonical variables derived from gravel and total Ni variations, with designation of sediment class identified by the cluster analysis (Figure 18).

in order to provide a visualization of the separation of the sediment classes. Class means and all cases--except where overlap occurs--are plotted. The plot reveals a good separation of the sediment classes: SANDS, MUDDY SANDS, and MUDS, with respect to Ni and gravel content (Figure 19b).

These results suggest that, with respect to sediment texture, Ni content has the greatest percent variation of any metal, and is a strong reflector of the mud fraction in the sediments.

Heavy Metal Fractionation Patterns

The raw data from the sequential extractions are listed in Appendix VIII, and summarized in Appendix XII. Data from Appendix XII were modified to elucidate the possible factors responsible for the metal-partitioning patterns observed between extractions. The first modification was to take the ratio of Group II data to the corresponding Group I data for each metal and station (Appendix IX). As you may recall, Group II metal fractions presumably represent the moderately stable, Fe oxide phases, while Group I metals are derived from the more "loosely held" phases such as exchangeable and carbonates. These ratios are shown in Table 10. A ratio of one would indicate equal portions of a metal's total sediment

concentration partitioned between Groups I and II. It is interesting, in light of the common belief that hydrous Fe oxides are efficient scavengers for trace metals in aquatic systems (Jenne, 1968; Lee, 1975), that none of the trace metals are nearly as strongly partitioned in the Fe phases (Group II), as is Fe itself (Table 10). Manganese and Cu in fact show no significant affinity for the Fe phases, based on the extraction methods adopted in this study (Table 10).

To check if any relationship exists between the metals and the carbonate contents of the sediments, the ratio of metals extracted into Step 2 (carbonate target, Table 5), to the carbonate contents measured on separate subsample portions from the corresponding stations (Appendix II-C), were tabulated in Table 10. Similar tabulations were done for Step 3 (organic complex target), versus organic carbon content. For carbonate, the lowest ratios occur where carbonate content is highest (e.g., in sediments at stations 13, 23, & 28), indicating the carbonate phase is not rich in metals, but may act to dilute the total-metal concentration in the sediment. For organic carbon, the stations with the highest organic-carbon contents, SL877-18 & UG-1, likewise, had generally the lowest ratios (Table 10). In sum, among all the variables analyzed to-date (Appendix II), only grain size seems to

TABLE 10

THE QUANTITIES OF HEAVY METALS IN IRON PHASES[v] VERSUS LEACHABLE FRACTIONS[w], IN CARBONATE PHASES[x] RELATIVE TO THE CARBONATE CONTENTS[y], AND IN ORGANIC PHASES[z] RELATIVE TO THE ORGANIC-CARBON CONTENTS[y] OF SIX SEDIMENTS FROM SIMPSON LAGOON

Station		V	Cr	Mn	Fe	Co	Ni	Cu	Zn
					{Group II/Group I}*				
	%Mud								
2	7	6.0	3.6	0.4	10.6	2.6	3.1	1.1	
13	92	7.6	12.0	0.5	15.2	3.9	4.7	1.6	
18	45	1.3	7.7	0.7	3.9	1.9	2.3	0.9	
23	75	3.5	4.4	0.6	13.3	2.4	2.6	1.6	
28	86	2.5	7.4	1.6	10.7	5.4	2.8	1.4	
UG- 1	44	3.8	4.9	0.7	13.4	3.1	1.4	1.4	
					{[Step 2]/$\%CO_3^=$}**				
	$\%CO_3^=$								
2	1.4	0.07	0.07	14.3	29	0.14	0.36	0.43	2.1
13	10.5	0.06	0.03	6.7	13	0.03	0.19	0.19	0.9
18	4.1	0.90	0.03	3.9	160	0.29	0.61	0.83	2.6
23	9.2	0.16	0.07	7.6	29	0.08	0.22	0.16	1.2
28	11.4	0.11	0.03	2.5	26	0.04	0.31	0.35	0.7
UG- 1	4.2	0.24	0.10	8.3	52	0.12	0.95	0.59	2.9

TABLE 10

(Continued)

Station	%OC	V	Cr	Mn	Fe	Co	Ni	Cu
		{[Step 3]/%OC}***						
2	0.2	1.3	0.20	133	1330	1.0	0.20	6.7
13	1.1	0.18	0.03	55	360	0.32	0.006	3.2
18	3.2	0.28	0.02	0.2	345	0.09	0.02	1.6
23	2.1	0.17	0.07	12	190	0.10	0.03	1.7
28	1.2	0.75	0.05	4.2	565	0.13	0.002	3.2
UG- 1	4.4	0.06	0.01	1.1	39	0.02	0.015	0.5

v Averages for Group II data from Appendix XII.
w Averages for Group I data from Appendix XII.
x Averages for Step 2 data from Appendix XII.
y Carbonate and organic carbon contents taken from Appendix II.
z Averages for Step 3 data from Appendix XII.

* The ratio of the quantities of metals extracted along with the Fe Phases (Group II) to metals extracted with the Leachable Fractions (Group I).

** The ratio of the quantities of metals extracted with the cation exchange resin (Step 2) to the carbonate content ($\%CO_3^{=}$) of the sediment at the corresponding station.

*** The ratio of the quantities of metals extracted with acetylacetone in benzene (Step 3) to the organic carbon content (%OC) of the sediment at the corresponding station.

clearly be a factor in the distribution of the eight metals in the sediments of Simpson Lagoon.

Results of Analysis of Variance on Heavy-Metal Fractions

The derived data for extraction Groups I and II were used in the ANOVAS on the results of the metal partitioning experiments, because the original seven fractions quantified were not sufficiently reproducible (Tables 6,7). Table 11 and 12 present the ANOVAS on extraction Groups I and II respectively.

The statistical significance of the F ratios obtained is indicated by the tail probability (P), which is the probability of having an F ratio greater than that listed, when sample means are the same (Snedecor & Cochran, 1980, pp. 220,221). This probability is very small, as shown, for most of the metals (Tables 11,12). For Fe and Cu in Group I data (Table 11), the P is about 20%, indicating that the reproducibility of the sum of Steps 1-4 (easily leachable phases) for these two metals is relatively poor, and the stations cannot be confidently discriminated. In general, however, the stations are different ($P < 0.0005$) with respect to the grouped extraction data, justifying the listing of individual station averages for the SES results (Appendix XII).

TABLE 11

ONE-WAY ANOVA ON THE INTER-STATION VERSUS THE INTRA-SUBSAMPLE SOURCES OF VARIATION, FOR THE AMOUNTS OF HEAVY METALS EXTRACTED WITH THE LEACHABLE FRACTIONS (Group I) OF SIX SEDIMENTS FROM SIMPSON LAGOON

Source of Variation	df	Sum of Squares	Mean Square	F Ratio	P*
VANADIUM					
Inter-station**	4_a	42.2_b	10.55^c	20.3	0.00^d
Intra-subsample	13^a	10.4^b	0.52		
CHROMIUM					
Inter-station**	4_a	2.4_b	0.59^c	6.2	0.012
Intra-subsample	9^a	1.5^b	0.10		
MANGANESE					
Inter-station**	4_a	56111_b	14028^c	38.3	0.00^d
Intra-subsample	13^a	6595^b	366		
IRON					
Inter-station**	4_a	$1097^{e}_{e,b}$	$274^{e,c}_{e}$	2.7	0.20
Intra-subsample	4^a	$1509^{e,b}$	100^e		
COBALT					
Inter-station**	4_a	1.1_b	0.28^c	3.0	0.07
Intra-subsample	11^a	1.7^b	0.09		

TABLE 11

(Continued)

Source of Variation	df	Sum of Squares	Mean Square	F Ratio	P*
		N I C K E L			
Inter-station**	4[a]	57.0[b]	14.26[c]	5.0	0.056
Intra-subsample	5[a]	51.3[b]	2.85		
		C O P P E R			
Inter-station**	4[a]	53[b]	13.21[c]	2.2	0.17
Intra-subsample	9[a]	101[b]	5.95		

* Approximate significance level, interpolated from the table of variance ratio F. This represents the probability of having an F ratio larger than that observed if the null hypothesis (that the station means are the same) holds.

** Inter-station variance calculated from the mean values of replicates from each station.

[a] Approximate df taken from Appendix XIV-A. This is a method to compensate for unequal variances between subsamples from different stations (Appendix XIII).

[b] Σ_1 from Appendix XIV-A.

[c] $n_h(s_{\bar{x}})^2$, where n_h = harmonic mean of unequal number of replicates for each station (Appendix XIII).

$(s_{\bar{x}})^2$ = variance of station means from Appendix X-L.

This is based on $s_{\bar{x}} = \frac{s}{\sqrt{n}}$, where:

$s_{\bar{x}}$ = standard deviation of the sample means.
s^x = standard deviation of the population.
n = sample size.

[d] Less than 0.0005.

[e] Multiply by 10^3.

TABLE 12

ONE-WAY ANOVA ON THE INTER-STATION VERSUS THE INTRA-SUBSAMPLE SOURCES OF VARIATION, FOR THE AMOUNTS OF HEAVY METALS EXTRACTED WITH THE IRON PHASES (Group II) OF SIX SEDIMENTS FROM SIMPSON LAGOON

Source of Variation	df	Sum of Squares	Mean Square	F Ratio	P*
VANADIUM					
Inter-station**	5_a	168.2_b	33.64^c	16.2	0.00^d
Intra-subsample	14^a	41.6^b	2.08		
CHROMIUM					
Inter-station**	5_a	64.8_b	12.96^c	15.4	0.00^d
Intra-subsample	12^a	16.9^b	0.84		
MANGANESE					
Inter-station**	5_a	20480_b	4096^c	39.0	0.00^d
Intra-subsample	8^a	2110^b	105		
IRON					
Inter-station**	5_a	$1996^e_{f,b}$	$399^{e,c}_f$	37.0	0.00^d
Intra-subsample	6^a	$1939^{f,b}$	108^f		
COBALT					
Inter-station**	5_a	22.4_b	4.49^c	13.6	0.00^d
Intra-subsample	11^a	6.0^b	0.33		

TABLE 12

(Continued)

Source of Variation	df	Sum of Squares	Mean Square	F Ratio	P*
		N I C K E L			
Inter-station**	5[a]	178.3[b]	35.65[c]	105	0.00[d]
Intra-subsample	12[a]	6.2[b]	0.34		
		C O P P E R			
Inter-station**	5[a]	180[b]	36.0[c]	6.4	0.009
Intra-subsample	9[a]	112[b]	5.6		

* Approximate significance level, interpolated from the table of variance ratio F. This represents the probability of having an F ratio larger than that observed if the null hypothesis (that the station means are the same) holds.

** Inter-station variance calculated from the mean values of replicates from each station.

[a] Approximate df taken from Appendix XIV-B. This is a method to compensate for unequal variances between subsamples from different stations (Appendix XIII).

[b] Σ_1 from Appendix XIV-B.

[c] $n_h(s_{\bar{x}})^2$, where n_h = harmonic mean of unequal number of replicates for each station (Appendix XIII).
$(s_{\bar{x}})^2$ = variance of station means from Appendix X-M.

This is based on $s_{\bar{x}} = \frac{s}{\sqrt{n}}$, where:

$s_{\bar{x}}$ = standard deviation of the sample means.
s^x = standard deviation of the population.
n = sample size.

[d] Less than 0.0005.

[e] Multiply by 10^5_4.

[f] Multiply by 10^4.

DISCUSSION

Distribution of Heavy Metals in the Sediments of Simpson Lagoon

Sediment texture is an important factor for the concentrations of heavy metals in nearshore sediments, as evidenced in many case studies (e.g., Williams et al., 1978; Hiraizumi et al., 1978; Mayer & Fink, Jr., 1980; Thorne & Nickless, 1981; Forstner & Wittmann, 1981, pp. 121-130; Tessier et al., 1982; Voutsinou-Taliadouri & Satsmadjis, 1982). Results of this investigation suggest that sediment texture may be the dominant factor in controlling the abundances of heavy metals in the sediments of Simpson Lagoon (Figure 17, Table 8). It would seem, based on the statistical analyses of this study, that the metal which is most affected by sediment texture in Simpson Lagoon is the total (HFNA) Ni (Table 9). Nickel's strong correlation with particle size can be understood in terms of changes in specific surface area and mineral composition, with changes in grain size.

As particle size diminishes, at least three sources of Ni in the sediment increase. First, the finer size fractions have greater specific surface areas with in-

creased adsorptive properties, and larger proportions of Fe-oxide coatings. It is therefore implied that Ni, either directly adsorbed to particle surfaces, or co-precipitated with Fe-hydroxide sols, would be expected to be relatively enriched in muddy sediments. Secondly, organic matter, which is usually more abundant in fine-grained sediments too, is also an important scavenger of trace metals (Nissenbaum & Swaine, 1976; Sholkovitz & Copland, 1981; Sohn & Hughes, 1981; Davis, 1984). Thirdly, the mineralogy is different in the fine fractions: there are generally smaller proportions of quartz and feldspars, which usually are poor hosts for heavy metals, but more secondary weathering products such as clay minerals. Clay minerals are relatively enriched in trace metals due to the substitution of the metals for major elements in their crystal lattices.

Naidu et al. (1982) have identified the presence of two major clay-mineral suites in Simpson Lagoon, and attributes the lateral variations in clay minerals to inputs of the clays from two major terrigenous sources: the Colville and Kuparuk Rivers. The statistical analyses based on 28 sediment variables (Figure 17, Table 8) accomplished in this study, however, have not shown any significant clustering of stations in Simpson Lagoon which compliment these two clay-mineral suites (cf. Figure 20). Since both

the heavy metals and clay minerals are concentrated in the mud fraction, it would seem by implication that the clay mineralogy of the <2μm fraction may not be a major factor for the concentrations of the heavy metals in the gross (HFNA) and extractable (HHAA) sediment fractions (cf. Figure 17).

Figure 20 in fact indicates a lack of any longitudinal trends across Simpson Lagoon for the three sediment types delineated by the cluster and discriminant analyses. Since total Ni, a heavy metal, is the best discriminator for the regional pattern shown in Figure 20 (Table 9), it is conceivable that the chemical composition of the sediments in western Simpson Lagoon is quite uniform, except for variations imposed by textural parameters. It is concluded that the statistical analyses based on the 28 combined textural, clay mineral, carbon, and heavy-metal variables, could not detect any longitudinal trends in the heavy-metal chemistry of Simpson Lagoon which may result from two different sources: the Colville and Kuparuk Rivers.

Analytical Constraints on the Heavy Metal Data

There are several factors between the time of sampling and the laboratory analysis, which can influence the credibility of any study of the concentrations and

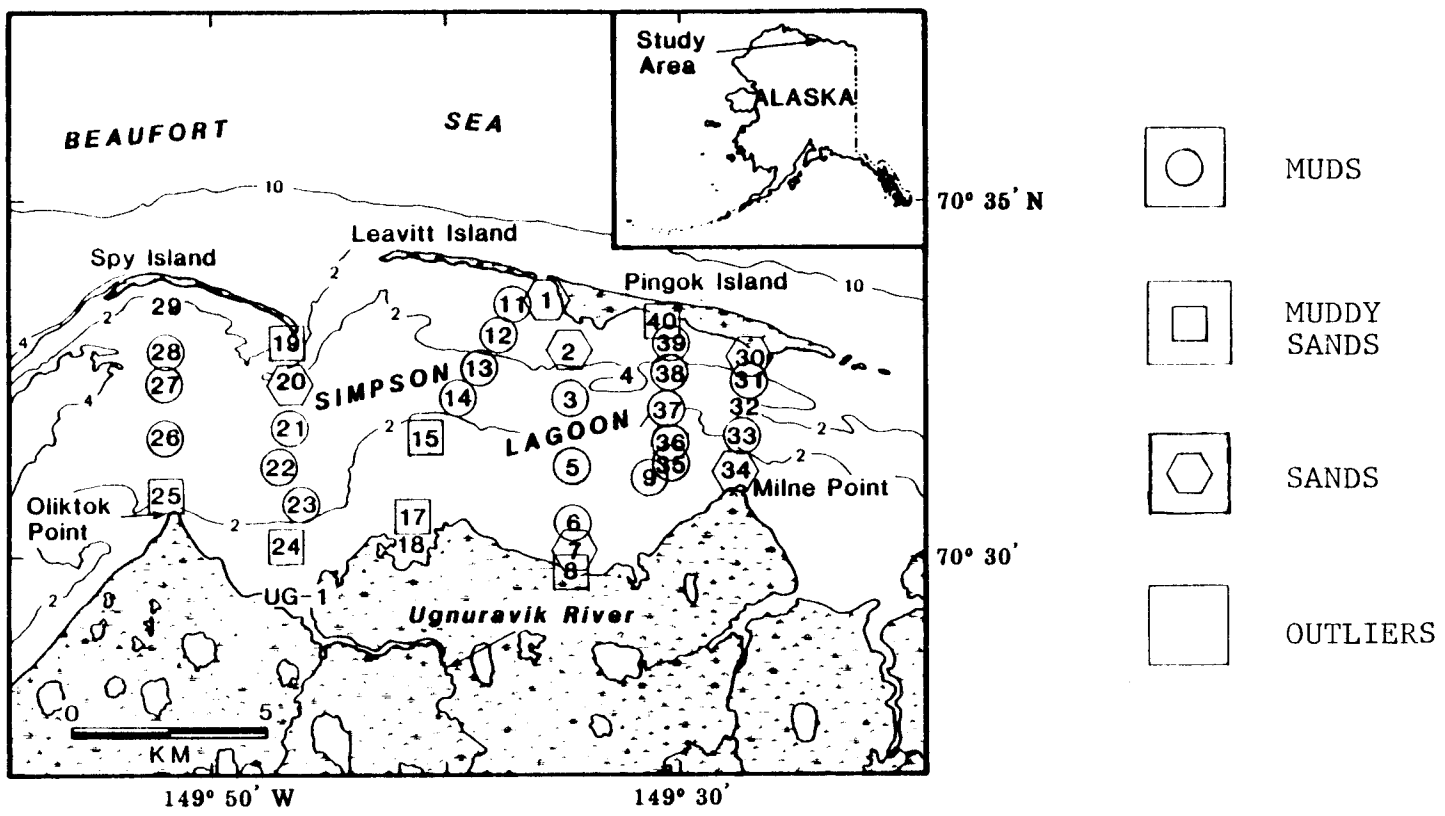

Figure 20. Classification of the sediment at each sampling location of this study in western Simpson Lagoon, according to results of the cluster and discriminant analyses on 28 variables.

speciations of metals in marine sediments. Some of these factors can be considered in the context of: sampling, sample preservation, sample preparation, and techniques adopted in the laboratory in an attempt to elicit the metal-partitioning patterns. Routine field and laboratory procedures currently in wide use, can in fact by design, introduce serious errors in geochemical investigations. For example, it has been observed that a wet, original sediment sample yields at least 20% more extractable Fe than its dried representative (Thomson, et al., 1980). In the context of the foregoing discussion, some of the questions relevant to the present study are: how accurate are the methods routinely adopted for estimating the total abundances of the eight metals in sediments, and do the chemical extraction procedures adopted herein provide reproducible results for the operationally-defined metal phases in the sediment? The implications of the results of this research are now discussed with respect to two specific questions:

a) what is the minimum number of samples that must be collected and analyzed for a given nearshore region, particularly in the Alaskan Arctic, to provide a representative estimate of the total and extractable (Chester and Hughes, 1967) amounts of heavy metals in sediments?

b) how many analytical replicates are necessary to obtain representative results for heavy metals released by each chemical extraction or digestion in this study?

The costs involved in the collection and analysis of a large number of marine sediment samples can pose a serious constraint to any pollution-monitoring program. In the nearshore, where a mosaic of lithologies are generally encountered, it would seem necessary to collect innumerable samples to represent the full range of sediment textures. Results of the statistical analyses (Table 9), however, suggest a sediment can be delineated into one of three classes with at least 95% confidence (not shown), based on its total Ni and gravel contents. This suggests one needs to take only three samples representing a sandy, muddy sand, and muddy sediment, to adequately characterize the heavy-metal abundances in sediments for a locality with a lithogical variability, and regional extent, similar to that of Simpson Lagoon.

The adoption of such a sampling strategy assumes that areal variations in mineral compositions, which could significantly modify the heavy-metal contents of the sediments, are relatively minor within such a local region, and that any anthropogenic inputs of metals are uniformly disseminated throughout the lagoon. It is also assumed that the redox conditions of the surficial sediments throughout the entire lagoon are similar; the anoxic sediment encountered in the restricted embayment of station 18 (an OUTLIER) is an exceptional case (Appendix I).

In a related context, there are other questions to be addressed. For a representative sample, what is the minimum number of replicate analyses necessary to precisely estimate heavy metals extracted by each chemical treatment? Results of this study have demonstrated that for most total (HFNA) and extractable (HHAA) metals, the dispersion in results is much less than 15% (Tables 6,7). This suggests that merely a single analysis on each sample will satisfactorily provide a precise estimate for the corresponding heavy-metal concentrations.

A set of at least three replicate sequences performed on each sample, however, would seem necessary to compensate for the much poorer reproducibilities obtained for some of the sequential treatments (Tables 6,7). The potential for loss or contamination of some metals during the complicated sequential extraction procedures (SES), appear to also severely impair their accuracies (Appendix XI). It is therefore suggested, that all the published data on the partitioning of metals in marine sediments, which do not document their corresponding accuracy and precision, must be accepted with caution. These reservations complement the conclusions of Guy et al. (1978), who criticized the lack of specificity and recovery efficiencies of so-called "selective" extraction reagents, basing their conclusions on experiments with model sediments.

Proposed Sequential Extraction Design

Several sequential extraction schemes (SES) have been proposed in attempting to elicit the partitioning patterns of metals in soils and sediments (e.g., Tessier et al., 1979; Forstner & Wittmann, 1981; Chao, 1984). These schemes have been criticized, however, because of their lack of specificity in defining the metal associations of sediments they purport to differentiate (Guy et al., 1978; Lion et al., 1982). Treatments designed to attack chemical phases with theoretically distinct stabilities, are likely to partially act on all the constituents in a sediment assemblage simultaneously, due to the intimate associations of the chemical phases. Thus some mobilization of metals into solution from metal fractions not targeted by a particular extraction step is likely.

The results of this study have demonstrated that the complex extraction designs popularly adopted by geochemists are also fraught with problems of precision and accuracy (Tables 6,7; Appendix XI), possibly related to the limitations of the reagents and procedures for specifying the chemical phases. Contributing to this situation is the ability of some sediment substrates to retain some of the metals which were mobilized during the chemical treatments, thereby reducing the recovery of metals associated with the destroyed phases. Guy et al.

(1978) and Rendell et al. (1980) have shown such readsorption of liberated metals during extractions with various reagents presently in common use.

Other analytical disadvantages inherent to SES are the numerous requirements for handling the samples and washing the associated glassware and labware. This naturally increases the chances of contamination or loss of recoverable metals (Appendix XI). Because of these theoretical and analytical problems, an eight-step SES for partitioning studies of heavy metals in sediments, is not recommended for use on "unpolluted" sediments such as those of Simpson Lagoon. The alternatives must be limited to extraction schemes which are less pretentious in the number of chemical fractions attempting to be discriminated.

The analytical strategies adopted in this study, however, provide an opportunity to compare an example of the simplest type of scheme: one treatment, e.g., Chester & Hughes reagent, HHAA; and one of the most complex: the eight-step sequence. It may be recalled that the precision for the amounts of metals extracted by most reagents in the eight-part scheme of this study was poor (Tables 6,7). The 50% to over 100% variability for metals recoverable by most of these treatments, may be partially compensated by replicating the experiments, but the

results, in terms of geochemical relationships of the metals, would still be highly questionable (Oakley et al., 1981). In this respect, single extraction techniques like the HHAA are more attractive, due to their superior precision.

Important additional information on the partitioning of metals between operationally-defined fractions, however, is provided by multi-extraction schemes relative to single leachings such as HHAA. For example, only a fraction (except for Mn) of the non-residual metals recovered by the eight-step scheme (Steps 1-7), are released by the HHAA method (Tables 6,7; Appendix XII). Additionally, HHAA does not appear to successfully extract metals associated with organic matter (e.g., Cu, also reported by Agemian & Chau, 1977), and only attacks a portion of the amorphous Fe oxides released by the oxalate treatment: Step 5 in Table 5 (Tables 6,7; Appendix XII). As implied previously, however, the eight-step design may at most be able to reproducibly discriminate between three derived fractions of the sediments: Groups I,II; residual (cf. Tables 6,7).

In light of the preceding discussion, it would seem reasonable to arrive at a compromise between the superior precision of single-extraction methods (e.g., HHAA), and the additional information gained about chemical frac-

tionations via the multiple extractions. A three-step extraction design is therefore proposed for nearshore oxic sediments, to estimate heavy metals associated with the following groups of chemical phases:

1) EASILY LEACHABLE:	Exchangeable, Carbonates, Organic complexes, Mn oxides
2) IRON PHASES:	Amorphous Fe oxides, Moderately reducible crystalline Fe oxides
3) RESIDUAL:	Silicates and Refractory oxides

As may be recalled, by the adoption of such an arbitrary grouping of sediment phases in this study, the analytical precision for heavy-metal fractionations dramatically improved (Tables 6,7). It is expected that this tri-fold extraction design would in fact obtain even better precision, since the cumulative errors associated with the repeated handling and analysis for the eight partial treatments would be decreased to errors associated with the procedures for only three treatments. A single experiment with this three-part sequence, performed on representatives of each sediment type (sand, muddy sand, and mud), should provide reproducible chemical-fractionation data for the metal distributions in a small area such as Simpson Lagoon.

The reagents I would recommend for this three-part scheme are as follows:

1) Easily Leachable--	COLD, 0.5 N HCl, 4 hours (Chester & Voutsinou, 1981)
2) Iron Phases--	0.04 M HHAA, 3 hours, 90° C, (Gupta & Chen, 1975)
3) Residual--	$HF-HNO_3$ (Rader & Grimaldi, 1961)

Regarding these proposed reagents, the dithionite treatment was replaced in step 2 with a popular, hot "hydroxylamine" (HHAA) technique, to avoid the problems of Zn contamination and sulfide precipitation of the former. The temperature and duration of steps 1 and 2 would have to be adjusted to maximize the discrimination between the relatively labile phases (1), and the more resistant iron oxides (2).

This scheme is applicable only to unpolluted, oxic sediments, where organic matter and sulfides are not quantitatively important as hosts of the metals. It is believed, based on the results of this study, that adoption of this three-part SES would provide both adequate precision, and the differentiation of three operationally-defined metal phases in arctic nearshore sediments: the cold-acid leachable--presumably more susceptible to remobilization processes; the Fe phases--which can be

mobilized under strong reducing conditions; and the residual minerals--which are largely refractory in the natural environment.

Comparison of the Concentrations of Heavy Metals in Nearshore Sediments of the Alaskan Beaufort Sea, and Those of the Tropical and Temperate Areas of the World

The average and maximum concentrations of Fe, Mn, Zn, V, Cr, Ni, Cu, and Co in gross sediments reported for Simpson Lagoon, Beaufort Lagoon (Figure 5), in the adjacent shelf of the Beaufort Sea, and a number of nearshore environments from around the world, are listed in Table 13. It must be emphasized that the data included in Table 13 pertain to gross metal concentrations, and shallow water, surficial, oxic, sandy, and unpolluted sediments, or to estimated background levels (surficial sediments) in areas impacted by heavy-metal pollution. The control of these variables is vital in order to meaningfully compare heavy-metal abundances in sediments from different regions; the influence of pollution, sediment texture, Eh, mineralogy, and sedimentation rates, must be taken into account (Table 14).

In recent years, many authors have compared their own data on heavy metals in nearshore sediments, to averages for nearshore Atlantic muds compiled in the early 60's by Wedepohl (1960). In these comparisons, however, the metal

TABLE 13

AVERAGE AND MAXIMAL NATURAL[a] CONCENTRATIONS (μg/g, except Fe which is 10^4 μg/g) OF TOTAL HEAVY METALS FOR SEDIMENTS FROM ALASKAN BEAUFORT SEA AND SELECTED COASTAL ENVIRONMENTS FROM THROUGHOUT THE WORLD

Location	Fe		Mn		Zn		V		Cr		Ni		Cu		Co		Total Method	Sediment Texture[b]	# of Samples	Depth Range (m)	References
	X	max	X	max	X	max	X	max	X	max	X	max	X	max	X	max					
ALASKAN ARCTIC COAST																					
Simpson Lagoon	2 0	2 9	270	550	75	120	72	100	47	70	23	32	17	33	7 5	10	HF HNO$_3$	S to SM	39	0 3	This study.
Beaufort Lagoon	2 6	3 5	350	470	70	100	100	140	58	95	21	29	20	28	12	18	HF HNO$_3$	S to SM	17	0 3	This study.
Beaufort Sea																					
Nearshore	2 1	3 8	310	650	80	170	90	160	53	100	29	49	20	35	8 5	16	HF HNO$_3$	MS to SM	65	0 10	This study.
Outer Shelf	2 8	4 9	390	690	90	160	135	290	77	125	40	70	27	55	13	30	HF HNO$_3$	SM	50	10 64	This study.
SOUTHERN CALIFORNIA																					
Los Angeles Harbor	1 6	2 9	250	410	50	80			30	50	18	31	18	25			HF HNO$_3$	CS to MS	8	0 15	Chen & Lu, 1974.
San Pedro Channel	1 7	2 3			45	70			30	50	23	30	14	25			HF HNO$_3$	S to SM	7	15 40	Chen & Lu, 1974.
EAST COAST NORTH AMERICA																					
Pamlico Sound																					
Jarret Bay	3 0	3 0	175	200			70	70	65	70	18	20	18	20	5	5	Emission Spec	SM	3	1 2	Berryhill *et al.*, 1972.
South River Estuary	2 7	5 0	500	700			40	70	30	70	10	15	60	100	7	7	Emission Spec	MS	4	0 4	Berryhill *et al.*, 1972.
Buzzards Bay, Mass	2 0	4 3	190	300	50	87	40	80	30	80	10	27	12	37	3	7	Emission Spec	CS to M	126	0 20	Moore, 1963.
Bay of Fundy, Nova Scotia	2 6	3 7	640	700	51	104	70	136	57	350	15	46	15	32	12	27	Total Digest	S to VSM	83	0 100	Loring, 1982.
Northern Placentia Bay	2 9	3 6	615	700	70	95			40	66	30	49	20	45	19	34	HF	S to MS	16	0 200	Wiley, 1976.
NORWAY																					
Framvaren Fjord	3 4	4 4	560	1000	87	100					19	23	11	18	18	24	HF H_2SO_4	S to M	6	1 14	Piper, 1971.
VENEZUELA																					
Sands	3 0	4 9	480	1100			73	135	30	53	17	23	7	13	7	11	Emission Spec	S to MS	15	0 20	Hirst, 1962.
Muds	5 1	6 3	1570	2900			130	150	86	99	30	34	18	23	12	13	Emission Spec	M to Cl	9	0 20	Hirst, 1962.
JAPAN																					
Nanao Bay	4 5	5 4	250	310	140	270					20	27	18	29	5	7	X ray Flour	Mud	19	5 45	Yamamoto *et al.*, 1977.
AUSTRALIA																					
Halifax, Cleveland Bays	1 9				31						11		7		7 5		HF HNO$_3$, HClO$_4$		56	5 20	Knauer, 1977.
Genoa River Estuary	4 3		180		74		94		51		24		13		15		X ray Flour	SM	42	0 6	Reinson, 1975.
MEAN VALUES	2 9	4 1	430	690	70	125	73	110	46	73	19	31	18	35	10	16					
AVERAGE NEARSHORE ATLANTIC SEDIMENTS[c]	4 8		850		95		130		100		55		48		13						

[a]Data from unpolluted sediments or natural background estimates were used.

[b]CS, S, MS, VSM, SM, M, Cl denote coarse sand, sand, muddy sand, very sandy mud, sandy mud and mud, clay, respectively.

[c]From Wedepohl, 1960.

fraction studied (total or extractable), the occurrence of pollutant components, or the sediment texture, are often not satisfactorily documented. This lack of reporting results of measurements on related sediment parameters limits the applicability of the corresponding metal data, as evidenced by the comparison of Wedepohl's (1960) averages with the recent compilation for sandy nearshore sediments made in this study (Table 13). The abundances of the eight metals in the sandy sediments are on average but 40% to 80% of their corresponding data for the Atlantic muds (Table 13). This implies that metal-abundance data in the future must better qualify sediment and environmental parameters in addition to water depth (nearshore), before the geochemistries of heavy metals in nearshore sediments from different regions of the world can be critically compared.

It is possible, however, to draw some tentative conclusions from Table 13, concerning the abundances of heavy metals in nearshore Beaufort Sea sediments relative to tropical and temperate regions. It seems that sediments of the nearshore Arctic of Alaska, have metal concentrations which fall within the natural ranges for nearshore sediments throughout the world. This suggests that the distinct weathering and depositional regimes of the Arctic may not be dominant factors in controlling the gross

heavy-metal abundances in the nearshore sediments. It probably also reflects a paucity of anthropogenic sources of these metals in the Alaskan Arctic.

Effects of Environmental Perturbations On the Abundances and Behaviors of Heavy Metals in the Nearshore Sediments of the Beaufort Sea

(a) determining the labile metal fractions in the sediments with the SES

Although the wet-chemical treatments popularly employed today are not as selective for sediment phases as often portrayed, they are one of the few tools available to examine the chemical associations of trace metals within natural particulate materials. SES are also useful in establishing bioavailabilities of sediment-bound metals (Jenne & Luoma, 1977; Luoma & Bryan, 1979; Luoma & Davis, 1983). Chemical extractions also provide a means to estimate the quantities of metals which may be released into the water column in response to perturbations in the physical or chemical conditions.

As suggested by the results of the SES on sediments from six stations of Simpson Lagoon, roughly 50% of all the metals except V and Cr (<20%), are partitioned in non-residual phases of the sediments: Steps 1-7 --those presumably not bound within the crystal lattices of

mineral particles (Appendix XII). These non-residual fractions of the metals, may potentially be released from the sediments due to such physico-chemical modifications of the sedimentary environment as pH, Eh, ionic strength, ligand concentrations, or hydraulic mixing (Table 15).

TABLE 14

FACTORS CONTROLLING HEAVY-METAL DISTRIBUTIONS IN SEDMENTS

Factor	Effect
High sedimentation rate and/or flux of organic matter	Active reduction in subsurface layers, with enrichment of some metals in surficial layers by diffusion of mobilized (reducible) metal phases
Heavy minerals, e.g., pyrite, ilmenite, magnetite, chromite	Relatively enriched in trace metals compared to predominate quartz, feldspars, and clay minerals
Industrial and domestic discharge of wastes enriched in metals	Supplementary flux of heavy metals added to natural flux from continental weathering
Proportion of silt and clay-size particles	Higher specific surface area and proportion of clay minerals, increases the abundance of adsorbed, hydrogenous, and lattice-held fractions of trace metals
Weathering regime	Specific geology of the hinterland controls the composition of detritus transported to the nearshore

It is important to note, based on the results of this investigation, that the quantities of metals which can be mobilized with relative ease from Simpson Lagoon sediments are considered to be very low (as represented by Group I treatments: exchangeable, carbonates, organic complexed, Mn oxides). These fractions (Group I) represent less than 15% of the total abundances for each metal, except Mn and Cu, for which it is about 30% (Steps 1-4 in Appendix XII). Only under strongly reducing conditions is it conceivable that the bulk of the Fe phases (Steps 5-7: Group II), can be mobilized from the sediment (Calmano & Forstner, 1983). This may be occurring naturally in at least one location in Simpson Lagoon: station 18 (Appendix I).

Highly polluted sediments in lower latitudes, which may contain over 1000 μg/g of some metals, would most likely have a deleterious impact on the associated ecosystem if the metals were remobilized. In the case of Simpson Lagoon, and presumably also its adjacent lagoons and nearshore areas, however, the 15% figure translates to only trace concentrations of a few μg/g for the metals (Appendices II-D,E). Intuitively, the mobilization of such low levels would not be a great threat to the ecosystem. Many biological systems may naturally exist on the margin of metal toxicity, however (Luoma, 1983).

It has been reported that natural concentrations of some trace metals in aquatic systems (e.g., Cu), can be toxic to some organisms, and any slight (µg/l) increases may have deleterious effects (Engel et al., 1981). Additionally, some of the less-stable Fe phases of Group II, in sediments routinely ingested by benthic fauna, may be habitually dissolved under the strongly acidic conditions prevailing in their guts. Intuitively, natural levels of these bioavailable fractions of metals, which are taken in during the normal feeding activities of such organisms, are either excreted, used to satisfy their nutritional requirements, or detoxified by nominal mechanisms they have had eons to evolve. The understanding of the possible ecological impacts of metals remobilized or supplied by sediments, must therefore take into account the observed magnitudes of the available metals, relative to species-specific tolerance levels.

Table 15 lists some of the possible natural and anthropogenic factors that may either trigger the release of metals from sediments into the overlying water, or supply additional sources of metal contaminants. The results of the present study on metal partitioning could conceivably offer a means to predict the proportions of the total metals that are susceptible to such remobilization. Some of the laboratory treatments on sediments

TABLE 15

NATURAL AND ANTHROPOGENIC FACTORS
WHICH INFLUENCE THE FLUXES OF HEAVY METALS
IN SEDIMENTARY ENVIRONMENTS

Natural or Anthropogenic Perturbations in the Physico-Chemical Conditions	Effects
1. Increase in salinity:	Desorption of metals by exchange with salt ions.
2. Change in redox conditions:	Dissolve oxides and hydroxides of Fe and Mn, and thus also mobilize associated metals.
3. Decrease in pH:	Dissolve carbonates, hydroxides of Fe and Mn, and exchange metals with hydrogen ions.
4. Hydraulic mixing of sediment:	Release pore waters into the overlying water.

****

Anthropogenic Disturbances	Effects
1. Dumping of dredge spoils:	Release of oxygen-consuming substances from reduced layers of sediment.
2. Dispersal of drilling muds:	Source of barium (Ba), Cr, Fe, and Zn.
3. Industrial and domestic effluents:	Source of metals and oxygen-consuming substances.
4. Emplacement of physical barriers, including docks, jetties, and causeways:	Alter the nearshore sediment budget in lagoons. This could lead to closing of inter-barrier channels and eventually restricted, stagnant waters.

adopted in this study, however, call for more drastic modifications of the Eh, pH, and ionic concentrations to mobilize metals from marine sediments, than are generally possible under natural or polluted conditions (i.e., extremely acidic or reducing conditions in treatments of Table 5). It is therefore implied that some of the conclusions drawn from the laboratory experiments herein, cannot be directly extrapolated without taking into account the lower reaction rates that are generally prevalent in the marine environment.

It would seem, however, that the results of the metal-fractionation studies could have a number of practical applications. The Simpson Lagoon and adjacent regions are ecologically sensitive areas (Johnson & J. W. Richardson, 1981), and concern has been expressed regarding the potential for oil spills resulting from the petroleum-related activities associated with the Prudhoe Bay, Kuparuk, and Milne Oil Fields adjacent to Simpson Lagoon. A subject of related context has been addressed in a case study concerning the impact of crude oil on heavy metals in intertidal sediments of subarctic Port Valdez (Naidu et al., 1978).

Naidu et al. (1978) demonstrated that the application of crude oil onto tidal-flat sediments resulted in a significant loss of Cu, Ni, and Zn from the sediments. The

proportions of metals thus mobilized were more or less equivalent to those released from the sediment in the laboratory under treatment with the Chester and Hughes (1967) reagent (HHAA). Based on their field and laboratory studies, Naidu et al. (1978) concluded that the mobilization of metals following sediment oiling, is effected by the onset of anaerobic conditions at the sediment surface resulting from the increased biological oxygen demand (BOD) of hydrocarbon-utilizing microorganisms. Oxygen levels presumably dropped, initiating reducing conditions, which released the metals originally stabilized in an oxidized sediment phase.

It is quite possible that such petroleum-induced mobilization of metals from sediments may someday also follow an oil spill in nearshore areas of north arctic Alaska. The amounts of metals thus mobilized may be ecologically significant, if indeed organisms can be affected by slight increases in the metal concentrations of seawater (Engel, et al., 1981). Additionally, Atlas (1977) has shown that oil disperses and weathers at a reduced rate in the cold, ice-covered environment of the Arctic, suggesting that any oil spilled in the nearshore of the Alaskan Arctic may persist for longer periods than in the temperate regions of the world. This implies that the reduction and remobilization of metals from sediments

exposed to oil may be relatively high in the arctic marine environment.

(b) discharge of drilling fluids

Fluids and muds associated with the drilling for petroleum reserves have begun to be discharged into the inner-shelf environment of the Beaufort Sea. One metal included in this study, Cr, is found in relatively high concentrations in these materials in the form of sodium chromate, chromic chloride, and ferrochrome and chrome lignosulfonates (Gettleson, 1980). These chromium compounds are predominantly non-residual, and thus potentially mobile. A comparison of the 5 μg/g levels of non-residual Cr in Simpson Lagoon sediments (Appendix XII), with the 300 μg/g concentrations in some discharges of drilling wastes (O.O.C., 1978 in Gettleson, 1980), indicates a contrast sufficient to permit detection of the dispersion of drilling wastes in the nearshore environments of the Beaufort Sea, by monitoring extractable Cr in the sediments. Although the Cr associated with drilling wastes may not prove to have a deleterious effect on the nearshore environment of the Beaufort Sea, its monitoring in sediments may be a simple means to follow the dispersal pathways of these materials.

(c) Ni and V in Prudhoe Bay crude

The Prudhoe Bay crude oil only contains about 10 μg/g Ni and 20 μg/g V (Clark and Brown, 1977). These concentrations are comparable to the extractable amounts in the sediments of Simpson Lagoon (Appendix XII). Only in a rare situation of a massive oil spill with a very concentrated, local accumulation of the associated V and Ni, is it conceivable to have levels detectable above background in the contaminated sediments. It is more likely that either the direct toxic effect of the hydrocarbons, or the decrease in the oxidation potential brought about following the degradation of the oil (would remobilize some metals), will be more responsible for any ecological damage.

Additionally, it is important to know the area of sediment rendered to a reduced state by a given quantity of oil, in light of comparing the amounts of metals mobilized from the sediment, with the amounts of metals introduced with the oil. With this knowledge, one possibly could determine the greater source of increased metal fluxes into the overlying seawater: the oil, or the sediment exposed to reducing conditions by the oil.

(d) dredging, resuspension, and redistribution of the sediments

With the increasing development occurring in the nearshore arctic environment of northern Alaska, including Simpson Lagoon, it is possible that sediments will be dredged to maintain or improve waterways or to supply materials for construction onshore. Heavy metals are frequently found in association with dredged material, and could conceivably be released during, and subsequent to dredging/disposal operations, thereby entering the marine food web (Wakeman, 1977; Tramontano, Jr. & Bohlen, 1984).

In the reducing conditions which occur in subsurface sediment layers, large fractions of the heavy metals originally bound in oxidative sediment precipitates, may get into dissolution and become a part of the interstitial waters. As a result, the subsurface sediment layers can serve as a potential reservoir for heavy metals in the dissolved state. Dredging operations could cause an almost instantaneous release into the overlying water of the metals held in the sediment pore waters.

Results of the present partitioning studies, however, indicate that at most, only small amounts (a few μg/g levels) of potentially-toxic trace metals such as Cu, Ni, and Cr, will be mobilized from sediment pore waters in the Alaskan Arctic, by the reworking processes associated with

dredging operations (Appendix XII). The possible effects of such small increases in fluxes of heavy metals to arctic ecosystems is not known. Perhaps laboratory experiments under controlled conditions, including specific, live arctic organisms exposed to different metal dosages, could provide data to predict the impacts of perturbations of heavy-metal fluxes on arctic marine ecosystems (Tatem, 1980).

SUMMARY

1) Total digestion with HF-HNO_3 was accomplished on gross sediment samples from 39 locations of Simpson Lagoon, in order to determine the heavy-metal concentrations. The average concentrations (µg/g) obtained were: Fe 20,000, Mn 250, and vary for the trace metals between Zn 75, and Co 8. These abundances generally fall within the natural ranges for sandy, nearshore sediments from many tropical and temperate regions. This suggests that the severe arctic climate may not be a major factor in the geochemistry of heavy metals in nearshore sediments, and probably reflects a scarcity of anthropogenic sources of metals in the Beaufort Sea. It follows, that the baseline data furnished by this study, can be used to monitor the adjacent marine environment for future heavy-metal contaminations from the ongoing petroleum-related activities in the Alaskan Arctic.

2) Sediment samples from all 39 stations were additionally subjected to a single treatment with

hydroxylamine hydrochloride-acetic acid reagent (HHAA), in order to mobilize the metals associated with the exchangeable phase (Exch), carbonates ($CO_3^=$), Mn oxides (Mnox), and some easily-reducible Fe oxides. Samples from six stations were also subjected to an eight-treatment, sequential extraction scheme (8tSES), to assess the HHAA results, and to furnish more detailed information on the partitioning of metals between operationally-defined sediment components. The respective targets of the 8tSES were Exch, $CO_3^=$, organic complexes (OrCx), Mnox, amorphous Fe oxides (aFeox), crystalline Fe oxides (cFeox), and residual/resistant minerals (Res). The comparison of the results from these two strategies (HHAA and 8tSES), indicates neither is to be recommended for routine partitioning studies of heavy metals in nearshore arctic sediments. The former, HHAA, does not discriminate well between the easily-leachable metal fractions (e.g., Exch, $CO_3^=$, OrCx, Mnox), and the moderately-reducible phases (aFeox, cFeox), as it simultaneously extracts metals from both. It should be noted, however, that because of materiel and procedural complexities, the 8tSES is not able to precisely differentiate the metal contents in the eight defined fractions of the sediments. A three-

treatment extraction scheme was therefore proposed whose precision is adequate, and appears to well-differentiate what appears to be three major fractions of metals in nearshore sediments of the Alaskan Beaufort Sea: Easily Leachable, Fe Phases, and Residual Minerals.

3) Metal-partitioning studies (e.g., HHAA, 8tSES), supply the environmental monitor with a means to predict or assess the fraction of total-metal contents which are susceptible to processes that can remobilize sediment-bound metals into overlying waters. They are also more sensitive to anthropogenic species of metals. Anthropogenic perturbations in the physical and chemical environment which may modify the fluxes of heavy metals in nearshore sediments include: the weathering of spilled petroleum, the discharge of domestic sewage or industrial wastes, the dredging of subsurface sediments, or a decrease in the supply of oxygen to the overlying water, due to a modification of circulation patterns by the introduction of causeways and jetties. Results of the 8tSES extractions indicate that <15% of the total-metal contents (except Cu, Mn, with 30%), of Simpson Lagoon sediments can be easily

mobilized, and 15% to about 50% may potentially be released from the sediments under strong reducing conditions (not generally occurring naturally in Simpson Lagoon). At the present time, however, these predictions are tentative, because the chemical reactions generated by most of the reagents used in this study, are not representative of the more modest fluctuations in pH and Eh normally encountered in nearshore environments. Additionally, it is not known what effect any remobilized metals may have on the resident biota of arctic lagoons.

4) The fact that heavy metals are indestructible elements at the Earth's surface makes them potential, long-term indicators of chronic, albeit low levels of general contamination. For example, it was suggested in this study, that the ongoing discharge into the Beaufort Sea of drilling wastes, related to the continuing search for offshore sources of petroleum, could be monitored by measuring the non-residual Cr fractions in the sediments, since Cr is a significant component in drilling wastes.

5) There are many factors which may influence heavy-metal abundances in surficial sediments, including grain size, sedimentation rate, OM content, terrigenous source, and inputs of anthropogenic wastes which are enriched in metals. Results of this study indicate that grain size is the dominant factor controlling the heavy-metal distributions of sediments within the local extent of western Simpson Lagoon. Any regional differences in heavy-metal abundances--due to distinctions in mineral compositions for detritus transported to the lagoon from its three major sediment sources: the Colville and Kuparuk Rivers, and the sediments eroding from the adjacent coastal bluffs--were not articulated from the seemingly dominant influence of variations in sediment texture, by the statistical methods (on 28 variables) employed in this study.

6) The sampling and analysis of nearshore sediments for chemical parameters is a difficult business, because of many environmental and analytical factors. These include macro to micro sample heterogeneities (e.g., lagoon to subsample), theoretical limitations for attempting to interface results of laboratory analysis

on collected samples with the real *in situ* chemical states, and the complexity of the materials (near-shore sediments are an intimate assemblage of many minerals and compounds with various stabilities). Results of this study indicate that the environmental chemist must make a compromise between analytical methods with high precision and ease of operation, and complicated procedures which are time-consuming and imprecise, but provide more detailed insight into the chemical states of the elements. In the context of chemical extraction methods, Guy et al., (1978) and others have shown that the complex nature of sedimentary materials, hampers the ability of SES to differentiate individual chemical phases. This study reveals too, that regardless of what fraction(s) of the metals are mobilized by partial treatments, it is difficult to obtain reproducible results for some of the minor fractions (e.g., OrCx: Step 3 in Table 5).

7) Regional studies of chemical parameters must always determine if samples from different sites can be discriminated, i.e., does the parameter being measured vary more within the sample, or throughout the entire study area? This is very important when looking for trends in regional data. Stations in

Simpson Lagoon could be discriminated by every metal fraction studied, except some of the minor phases of the 8tSES. The adoption, however, of the recommended, three-step extraction sequence, most probably would provide adequate precision to discriminate stations in arctic lagoons in terms of three chemical fractions: Easily Leachable, Fe Phases, and Residual Minerals.

RECOMMENDATIONS FOR FURTHER RESEARCH

I With respect to the concerns expressed in the discussion section, it is recommended that the abundances of metals in nearshore sediments from diverse regions of the world, be quantitatively compared using statistical methods such as ANOVA. For each locality, along with the metal data, the environmental and sedimentary parameters which influence the metal distributions should be carefully documented, to provide an understanding for any observed differences. These parameters include sedimentation rates, the nature of the terrigenous source (gross mineralogy), climatic variables, oxidation potentials at the sediment/seawater interface, sediment texture, and pollutant fluxes.

II Much work needs to be done to better understand the behaviors and effects of the naturally- and potentially-labile metals in nearshore sediments. One solution may be to conduct laboratory and microcosm experiments with model and natural sediments (pristine and polluted), with concern for the

kinetics of mobilization reactions relative to variations in pH, Eh, and salinity conditions (e.g., Gambrell et al., 1980).

Similar experiments should also be performed with organisms, to aid in understanding the uptake and states of bioactive metal species. Strategies would include varying the biological species and the experimental conditions (e.g., pH), and then quantifying the metal fractions mobilized into the aqueous phase, or either temporarily or permanently accumulated by the organisms.

III Most studies on heavy metals in nearshore sediments merely provide survey data taken at a single instance, and are not concerned with the environmental dynamics. Clearly, to really understand the metal ecologies, the budgets for the cycling of the metals between the atmosphere, biota, suspended particles, bottom sediments, and waters should be determined. Periodic sampling in different seasons, and measurements of the metal fluxes across the sediment/seawater interface are specific examples to elucidate some aspects of the temporal and spatial variations of heavy metals in nearshore sedimentary environments.

APPENDIX I

SIMPSON LAGOON STATION INFORMATION

APPENDIX I

INFORMATION ON SEDIMENT GRABS RETRIEVED AT 1977 SIMPSON LAGOON STATIONS

Station	Date Collected	Minutes of Lat. 70°N	Minutes of Long. 149°W	R*	Water Depth (m)	Type of Sediment Observed**
SL877- 1	8/03	33.70	36.30	5	1.5	ms
SL877- 2	8/03	32.85	34.75	-	1.8	s
SL877- 3	8/03	32.05	34.65	-	2.6	sm
SL877- 4	8/03	32.61	32.55	-	2.1	sm
SL877- 5	8/04	31.10	34.85	4	1.8	fsm
SL877- 6	8/04	30.46	34.60	-	2.0	sm
SL877- 7	8/04	29.98	34.85	-	0.9	ms + gr
SL877- 8	8/04	29.83	34.90	-	0.3	ms
SL877- 9	8/04	31.13	31.30	-	1.5	ms
SL877-10***	8/04	30.82	31.30	-	1.5	s
SL877-11	8/06	33.55	37.50	-	2.1	m
SL877-12	8/06	33.12	38.05	-	2.1	m
SL877-13	8/06	32.60	38.95	-	2.4	m
SL877-14	8/06	32.02	40.45	-	2.3	m
SL877-15	8/06	31.40	41.15	-	1.8	m
SL877-16†	8/06	30.87	41.35	-	0.9	gr s
SL877-17	8/06	30.36	41.60	-	0.6	bms/H_2S
SL877-18	8/06	30.03	41.53	-	0.5	ms/H_2S [tundra mats]
SL877-19	8/07	33.00	46.80	-	3.2	ms
SL877-20	8/07	32.48	46.70	-	2.6	ms
SL877-21	8/07	31.78	46.85	4	2.9	m
SL877-22	8/07	31.02	47.20	-	2.4	dbm
SL877-23	8/07	30.50	46.50	-	2.4	om
SL877-24	8/07	29.95	46.85	-	1.5	ms
SL877-25	8/07	31.33	51.69	3	2.6	ms
SL877-26	8/07	31.58	51.85	-	2.9	m
SL877-27	8/07	32.23	52.01	-	3.0	Cm
SL877-28	8/07	32.92	52.20	-	2.7	m
SL877-29	8/07	33.57	52.35	-	2.4	m/pocht
SL877-30	8/14	32.72	26.80	-	1.1	ms
SL877-31	8/14	32.46	26.91	-	2.6	mm
SL877-32	8/14	31.96	27.20	4	2.3	sm
SL877-33	8/14	31.53	27.45	-	2.1	ms
SL877-34	8/14	31.18	27.60	-	1.5	s
SL877-35	8/14	31.18	30.81	-	2.0	sm
SL877-36	8/14	31.62	30.80	-	2.1	sm
SL877-37	8/14	32.16	30.72	-	2.3	m
SL877-38	8/14	32.55	30.78	-	2.3	m

APPENDIX I

(Continued)

Station	Date Collected	Minutes of Lat. 70°N	Minutes of Long. 149°W	R*	Water Depth (m)	Type of Sediment Observed**
SL877-39	8/14	32.93	30.58	-	2.0	m
SL877-40	8/14	33.36	30.55	-	1.5	ms
UG- 1	8/07	29.33	46.45	-	0.8	om

* Number of replicates.

** Explanation of Abbreviations: ms: muddy sand, s: sand, sm: sandy mud, fsm: fine sandy mud, gr: gravel, m: mud, gr s: gravelly sand, H_2S: H_2S odor present, bms: black muddy sand, dbm: dark brown mud, om: organic-rich mud, Cm: Colville River mud, pocht: polychaetes, mm: pure mud.

*** Sample not retrieved.

† Sediment from this station not analyzed in this study.

APPENDIX II

TEXTURAL, CLAY-MINERALOGICAL, AND CHEMICAL DATA
FOR SEDIMENTS FROM 39 STATIONS (inter-station variance)
OF SIMPSON LAGOON

APPENDIX II-A

WEIGHT PERCENTS OF GRAIN-SIZE FRACTIONS
IN SEDIMENTS OF SIMPSON LAGOON (SL877)

Station	Gravel	Sand	Silt	Clay	Mud*
1a	0.4	80.2	14.5	4.9	19.4
2	0.4	92.6	3.3	3.7	7.0
3	0.4	34.5	45.2	19.9	65.1
4	0.3	19.1	56.5	24.1	80.6
5a	0.0	35.2	52.8	12.0	64.8
6	0.1	32.1	50.5	17.4	67.9
7	17.1	75.0	3.7	4.2	7.9
8	9.1	65.1	17.3	8.5	25.8
9	0.0	24.9	54.8	20.3	75.1
11	1.3	14.7	65.7	18.3	84.0
12	0.7	29.9	52.6	16.8	69.4
13	0.0	7.7	79.5	12.8	92.3
14	0.2	38.1	47.6	14.1	61.7
15	5.0	69.9	13.0	12.1	25.1
17	14.0	61.7	15.0	9.3	24.3
18	0.3	54.4	31.0	14.3	45.3
19	1.6	65.8	23.4	9.2	32.6
20	0.0	85.2	9.0	5.8	14.8
21a	1.2	15.8	59.6	23.4	83.0
22	0.0	11.0	75.4	13.6	89.0
23	2.2	22.4	61.5	13.9	75.4
24	7.0	64.9	18.9	9.2	28.1
25a	1.3	62.5	26.1	10.1	36.2
26	0.0	20.2	66.8	13.0	79.8
27	0.1	19.8	64.2	15.9	80.1
28	0.0	14.3	64.9	20.8	85.7
29	3.1	19.2	57.3	20.4	77.7
30	2.2	76.0	18.1	3.7	21.8
31	0.0	10.5	64.2	25.3	89.5
32a	0.0	32.6	48.3	19.1	67.4
33	0.0	49.2	34.0	16.8	50.8
34	0.3	98.0	0.7	1.0	1.7
35	0.2	44.0	39.4	16.4	55.8
36	0.0	39.4	44.0	16.6	60.6
37	0.0	37.7	47.6	14.7	62.3
38	0.8	25.4	53.8	20.0	73.8
39	5.5	29.9	43.1	21.5	64.6
40	4.8	64.4	17.3	13.5	30.8
UG- 1	0.0	56.5	28.5	15.0	43.5

* Mud equals silt plus clay.

APPENDIX II-B

STATISTICAL GRAIN-SIZE PARAMETERS* FOR SEDIMENTS FROM SIMPSON LAGOON (SL877)

Station	Median Size Md(Ø)	Mean Size M_z(Ø)	Sorting σ_I(Ø)	Skewness Sk_I	Kurtosis K_G
1a	2.3	2.8	1.6	0.73	3.70
2	1.8	1.7	1.2	0.27	3.25
3	4.9	5.4	3.2	0.25	0.85
4	5.6	6.2	2.9	0.28	1.15
5a	4.6	4.9	2.3	0.34	1.40
6	4.6	5.2	2.8	0.31	1.20
7	2.1	1.2	2.4	-0.37	2.10
8	2.7	3.0	2.9	0.22	2.20
9	5.5	5.9	2.9	0.27	1.20
11	5.1	5.9	2.7	0.41	1.60
12	4.8	5.4	2.7	0.38	1.55
13	5.0	5.5	1.9	0.52	2.05
14	4.5	4.9	2.7	0.26	1.20
15	2.3	3.5	3.2	0.55	2.00
17	2.2	2.7	3.3	0.26	2.60
18	3.4	4.5	2.7	0.65	1.20
19	2.7	3.5	2.0	0.82	1.50
20	2.5	2.7	1.2	0.71	6.80
21a	5.6	6.3	2.8	0.36	1.15
22	5.0	5.6	2.0	0.52	1.75
23	5.1	5.1	2.7	0.10	1.70
24	2.5	3.4	3.1	0.35	2.00
25a	2.4	3.5	2.6	0.71	1.05
26	4.9	5.4	2.1	0.46	1.60
27	5.0	5.5	2.4	0.40	1.50
28	5.4	6.1	2.5	0.48	1.15
29	5.3	5.8	3.0	0.25	1.30
30	2.0	2.5	1.7	0.45	1.30
31	6.0	6.6	2.6	0.33	1.15
32a	5.3	5.3	3.0	0.15	0.85
33	4.3	4.8	3.0	0.34	0.85
34	2.0	2.0	0.6	0.13	1.00
35	4.7	5.1	2.7	0.35	0.98
36	4.8	5.1	2.8	0.25	0.85
37	4.7	4.9	2.7	0.27	1.00
38	5.1	5.4	3.1	0.19	1.05
39	5.0	5.2	3.7	0.07	1.05
40	2.5	3.7	3.1	0.59	1.90
UG- 1	3.4	4.4	3.0	0.55	1.40

* After Folk and Ward, 1957.

APPENDIX II-C

RELATIVE PROPORTIONS* OF CLAY MINERALS IN THE <2μm SEDIMENT FRACTION**, AND WEIGHT PERCENTS OF CARBONATE ($CO_3^=$) AND ORGANIC CARBON[Y], IN GROSS SEDIMENTS OF SIMPSON LAGOON (SL877)

STATION	CLAY MINERALS				CARBON CONTENT	
	Expandable Minerals	Illite	Kaolinite	Chlorite	Carbonate ($CO_3^=$)	Organic Carbon
1a	7	65	9	19	3.8	0.26
2	5	67	11	17	1.4	0.15
3	7	62	11	20	9.8	1.10
4	4	65	9	21	8.6	1.80
5a	7	63	10	20	10.3	0.58
6	8	59	10	23	9.5	2.50
7	2	71	10	17	6.0	0.22
8	2	66	11	21	5.3	1.80
9	4	63	12	21	9.4	2.30
11	10	59	12	19	11.0	0.50
12	8	62	10	20	10.0	1.60
13	14	54	10	22	10.5	1.10
14	3	66	12	19	7.8	1.30
15	5	62	12	21	2.6	0.31
17	2	65	13	20	7.6	0.54
18	4	66	9	21	4.1	3.20
19	6	64	10	20	5.9	0.40
20	10	57	12	21	1.9	0.36
21a	9	61	11	19	9.5	1.50
22	10	59	11	20	10.7	1.50
23	3	66	11	20	9.2	2.10

APPENDIX II-C

(Continued)

STATION	CLAY MINERALS				CARBON CONTENT	
	Expandable Minerals	Illite	Kaolinite	Chlorite	Carbonate ($CO_3^{=}$)	Organic Carbon
24	6	63	11	20	4.7	0.81
25a	11	59	11	19	4.8	1.70
26	13	55	11	21	9.5	1.20
27	13	55	11	21	10.4	1.20
28	7	57	13	23	11.4	1.20
29	5	68	11	16	10.5	0.70
30	2	67	11	20	3.1	0.33
31	8	59	10	23	11.8	1.60
32a	6	70	8	16	12.6	1.40
33	3	62	12	23	7.7	1.00
34	0	64	13	23	3.3	0.08
35	12	56	11	21	9.6	1.70
36	10	57	11	22	8.6	1.50
37	8	63	8	21	8.7	1.10
38	9	56	12	23	8.8	0.96
39	10	58	10	22	8.2	1.30
40	4	64	12	20	3.6	0.44
UG- 1	6	66	9	19	4.2	4.40

* Weighted peak area percentages, after Biscaye (1965).
** Identifies only four minerals in the clay-size fraction of the sample.
y Organic carbon equals the results of total carbon minus carbonate-carbon analyses on separate subsamples.

APPENDIX II-D

CONCENTRATIONS (μg/g, except iron which is in 10^4 μg/g) OF VANADIUM, CHROMIUM, MANGANESE, AND IRON IN SIMPSON LAGOON SEDIMENTS (SL877), RELEASED INTO HYDROFLUORIC-NITRIC ACID DIGESTS* (HFNA) AND HYDROXYLAMINE HYDROCHLORIDE-ACETIC ACID EXTRACTS** (HHAA)

STATION	VANADIUM		CHROMIUM		MANGANESE		IRON	
	HFNA	HHAA	HFNA	HHAA	HFNA	HHAA	HFNA	HHAA
1a	43	1.9	23	0.2	118	48	1.13	C.112
2	36	2.0	21	0.4	146	80	0.95	0.110
3	84	5.2	59	1.8	419	240	2.51	0.332
4	101	7.3	70	2.0	554	365	2.92	0.465
5a	69	3.6	39	1.5	273	155	1.82	0.228
6	75	4.9	54	1.7	369	218	2.30	0.307
7	45	1.6	20	0.5	150	45	1.22	0.039
8	61	2.7	32	1.0	200	125	1.33	0.120
9	80	5.2	55	2.1	514	340	2.48	0.301
11	84	5.9	56	1.7	268	122	2.34	0.262
12	81	4.7	54	0.9	278	136	2.39	0.250
13	79	5.1	60	1.8	467	300	2.64	0.370
14	73	4.2	47	1.3	361	208	2.26	0.270
15	61	3.4	38	0.7	226	137	1.67	0.095
17	49	3.3	33	1.2	176	110	1.31	0.140
18	74	6.7	54	1.7	163	60	1.93	0.410
19	61	2.0	36	0.4	185	64	1.75	0.147
20	43	2.5	22	0.4	159	80	1.31	0.156
21a	100	6.5	66	1.2	370	193	2.81	0.355
22	85	4.0	55	1.4	302	138	2.59	0.263
23	70	4.4	56	1.2	378	215	2.09	0.248

APPENDIX II-D

(Continued)

STATION	VANADIUM		CHROMIUM	
	HFNA	HHAA	HFNA	HHAA
24	58	2.8	33	0.3
25a	57	3.1	37	0.9
26	87	4.3	57	1.2
27	83	4.8	57	1.5
28	93	5.1	62	1.8
29	85	5.6	63	1.5
30	29	1.2	24	0.0
31	97	6.2	67	1.5
32a	97	5.0	52	1.5
33	63	3.5	45	1.4
34	35	1.1	16	0.0
35	85	4.3	54	0.9
36	88	4.7	54	1.1
37	82	3.2	50	1.0
38	95	5.1	60	1.1
39	85	5.2	63	1.0
40	63	2.1	38	0.1
UG- 1	56	3.5	46	0.7

* Rader and Grimaldi, 1961, p. A32.
** Chester and Hughes, 1967.

MANGANESE		IRON	
HFNA	HHAA	HFNA	HHAA
154	62	1.36	0.085
188	78	1.72	0.172
375	198	2.69	0.285
295	136	2.47	0.256
291	114	2.67	0.274
291	123	2.59	0.326
127	66	0.88	0.086
313	160	2.63	0.300
299	198	2.25	0.269
323	182	1.65	0.179
115	46	0.85	0.032
247	110	2.06	0.199
256	112	2.32	0.253
226	89	2.16	0.206
217	82	2.26	0.196
225	80	2.20	0.248
174	73	1.80	0.084
211	90	1.65	0.150

APPENDIX II-E

CONCENTRATIONS (μg/g) OF COBALT, NICKEL, COPPER, AND ZINC IN SIMPSON LAGOON SEDIMENTS (SL877), RELEASED INTO HYDROFLUORIC-NITRIC ACID DIGESTS* (HFNA) AND HYDROXYLAMINE HYDROCHLORIDE-ACETIC ACID EXTRACTS** (HHAA)

STATION	COBALT		NICKEL		COPPER		ZINC	
	HFNA	HHAA	HFNA	HHAA	HFNA	HHAA	HFNA	HHAA
1a	4.3	0.7	11	0.9	8	1.9	34	5
2	3.6	0.7	11	1.0	11	1.1	28	3
3	7.6	2.7	29	4.4	17	2.6	94	15
4	8.2	3.5	32	5.3	28	4.8	120	21
5a	6.2	1.9	22	3.3	14	2.4	78	11
6	7.9	2.3	28	5.1	23	2.8	84	16
7	5.5	0.6	13	1.0	11	2.6	33	4
8	6.8	1.0	19	3.8	12	1.5	52	6
9	9.2	2.9	29	5.5	27	3.7	91	18
11	8.5	2.1	29	3.9	18	3.1	87	17
12	9.8	2.5	29	3.8	20	3.0	83	16
13	9.2	3.0	29	5.0	23	3.5	88	18
14	8.9	2.6	26	3.8	17	2.6	78	12
15	9.0	1.9	20	3.2	15	3.6	61	6
17	5.6	2.7	20	5.2	14	2.3	43	11
18	8.7	3.1	27	6.1	20	2.3	78	22
19	5.7	1.0	19	1.2	16	3.5	86	11
20	4.8	1.3	11	0.9	9	1.9	42	6
21a	9.6	3.3	31	4.7	22	3.5	100	19
22	9.6	2.6	28	4.0	19	2.5	89	14
23	8.4	2.7	27	4.7	21	2.7	93	15

APPENDIX II-E

(Continued)

STATION	COBALT		NICKEL		COPPER		ZINC	
	HFNA	HHAA	HFNA	HHAA	HFNA	HHAA	HFNA	HHAA
24	6.1	0.9	16	1.4	12	2.3	51	10
25a	6.7	1.8	17	2.7	12	2.3	64	11
26	9.3	3.1	29	4.9	18	3.0	88	15
27	8.8	2.5	29	4.7	15	2.8	79	15
28	9.3	2.3	32	4.3	23	2.8	100	16
29	10.0	2.3	27	4.1	25	4.0	98	18
30	3.6	0.5	12	0.7	6	1.7	33	3
31	9.5	2.6	32	5.0	14	3.2	100	22
32a	8.4	2.7	27	6.1	16	3.1	93	23
33	7.6	2.3	25	4.1	18	2.3	74	14
34	3.3	0.3	8	0.2	7	1.4	31	3
35	7.4	1.8	26	3.7	19	2.8	85	16
36	8.4	2.3	27	3.7	21	2.7	92	16
37	8.4	1.7	25	3.0	18	2.3	81	13
38	8.8	2.2	27	4.3	20	2.7	83	18
39	8.8	1.9	28	3.4	33	3.9	84	18
40	6.6	1.0	18	2.0	10	2.5	68	6
UG- 1	6.0	2.1	21	4.0	16	0.4	65	12

* Rader and Grimaldi, 1961, p. A32.
** Chester and Hughes, 1967.

APPENDIX III

RESULTS OF REPLICATE ANALYSES FOR GRAIN-SIZE PARAMETERS ON SIMPSON LAGOON SEDIMENTS

APPENDIX III-A

RESULTS OF GRAIN-SIZE ANALYSIS ON REPLICATE SEDIMENT GRABS (intra-station variance) RETRIEVED FROM FIVE STATIONS IN THIS STUDY (SL877)

Station	Grav. (%)	Sand (%)	Silt (%)	Clay (%)	Mud (%)	Md (ø)	M_z (ø)	σ_I (ø)	Sk_I	K_G
1a	0.40	80.22	14.50	4.88	19.38	2.26	2.83	1.57	0.73	3.72
1b	0.30	88.65	6.58	4.47	11.05	2.24	2.27	1.15	0.42	4.80
1c	-	86.93	8.17	4.90	13.07	2.27	2.32	1.22	0.45	5.00
1d	0.09	83.27	10.16	6.49	16.65	2.30	2.84	1.87	0.73	5.91
1e	0.08	82.49	11.96	5.47	17.43	2.29	2.82	1.71	0.69	4.82
5a	-	35.19	52.77	12.04	64.81	4.58	4.90	2.30	0.34	1.39
5b	-	48.96	37.14	13.90	51.04	4.05	4.73	2.50	0.50	1.40
5c	0.55	43.91	41.93	13.61	55.54	4.27	4.89	2.40	0.49	1.29
5d	1.68	47.26	38.58	12.48	51.06	4.05	4.72	2.41	0.50	1.49
21a	1.24	15.79	59.55	23.42	82.97	5.59	6.28	2.78	0.36	1.16
21b	0.33	16.13	61.90	21.64	83.54	5.61	6.15	2.68	0.32	1.19
21c	-	22.11	59.49	18.39	77.88	5.20	5.60	2.63	0.29	1.25
21d	2.82	15.12	62.64	19.42	82.06	5.37	5.88	2.74	0.31	1.32
25a	1.28	62.52	26.13	10.07	36.20	2.39	3.53	2.64	0.71	1.04
25b	0.37	66.44	24.79	8.39	33.18	2.29	3.42	2.66	0.72	1.16
25c	0.37	61.54	27.13	10.96	38.09	2.62	3.74	2.72	0.67	1.03
32a	-	32.61	48.34	19.05	67.39	5.27	5.35	2.98	0.15	0.84
32b	-	16.59	58.92	24.50	83.42	6.45	6.43	2.81	0.02	1.22
32c	-	26.25	53.55	20.19	73.74	5.63	5.61	3.05	0.11	0.96
32d	-	33.57	48.48	17.95	66.43	5.37	5.32	2.95	0.10	0.83

APPENDIX III-B

RESULTS OF GRAIN-SIZE ANALYSIS ON REPLICATE 50-GRAM PORTIONS (intra-sample variance) OF SINGLE SEDIMENT GRABS FROM THREE STATIONS IN THIS STUDY (SL877)

Station	Portion	Grav. (%)	Sand (%)	Silt (%)	Clay (%)	Mud (%)	Md (Ø)	M_Z (Ø)	σ_I (Ø)	Sk_I	K_G
15	1	5.04	69.88	12.97	12.11	25.08	2.28	3.46	3.20	0.55	2.01
	2	4.84	70.43	13.63	11.10	24.73	2.27	3.39	3.06	0.56	2.16
	3	2.67	72.87	13.22	11.23	24.45	2.27	3.38	2.82	0.68	2.08
31	1	-	10.52	64.19	25.29	89.48	6.00	6.61	2.65	0.33	1.14
	2	-	11.07	60.00	28.93	88.93	6.67	6.97	2.70	0.18	1.10
	3	-	11.03	59.92	29.06	88.98	6.70	6.98	2.58	0.16	1.13
33	1	-	74.89	17.04	8.07	25.11	2.37	3.54	2.33	0.80	1.63
	2	-	80.55	12.65	6.80	19.45	2.32	3.32	2.14	0.78	3.74

APPENDIX IV

RESULTS OF REPLICATE ANALYSES FOR CLAY MINERALS IN THE LESS THAN 2 μm FRACTION OF SEDIMENTS FROM SIMPSON LAGOON

APPENDIX IV-A

RELATIVE PERCENTAGES OF CLAY MINERALS IN REPLICATE SEDIMENT GRABS (intra-station variance) RETRIEVED FROM FIVE STATIONS IN THIS STUDY (SL877)

Station	Expandable Minerals	Illite	Kaolinite	Chlorite
1a*	6.7	65.0	9.3	19.0
1b	9.0	58.4	10.6	22.0
1c**	9.9	59.1	9.3	21.7
1d	9.2	62.7	8.9	19.2
1e	8.1	61.2	8.2	22.5
5a*	6.4	63.1	10.2	20.3
5b	8.5	59.6	10.5	21.4
5c	7.7	59.3	9.0	24.0
5d	6.7	63.3	10.3	19.7
21a	8.6	60.9	10.8	19.7
21b***	12.7	56.8	10.0	20.5
21c	5.0	67.6	10.1	17.3
21d*	4.6	67.8	11.1	16.5
25a	11.0	59.3	11.0	18.7
25b*	11.3	58.8	10.3	19.6
25c	12.2	56.6	10.8	20.4
32a	5.4	70.3	8.4	15.9
32b*	4.5	68.0	8.1	19.4
32c	6.8	60.7	10.6	21.9
32d*	9.7	56.5	10.0	23.8

* Average values from Appendix IV-C.
** Average values from Appendix IV-D.
***Average values from Appendix IV-B.

APPENDIX IV-B

RELATIVE PERCENTAGES OF CLAY MINERALS IN SEPARATE DISPERSIONS* (intra-sample variance) OF FOUR 50-GRAM PORTIONS OF THE SEDIMENT GRAB FROM STATION SL877-21b

	Clay Mineral			
Dispersion	Expandable Minerals	Illite	Kaolinite	Chlorite
1**	15.5	54.4	9.3	20.8
2***	11.0	59.0	10.3	19.7
3	12.5	56.5	10.2	20.8
4**	11.8	57.4	10.4	20.4

* The <62μm fraction (mud) of each 50-gram sample split is dispersed in one liter of distilled water. A small fraction of the <2μm particles in this dispersion is collected and made into a dense water-sediment slurry. A portion of this slurry is then mounted on a slide.

** Average values from Appendix IV-C.

*** Average values from Appendix IV-D.

APPENDIX IV-C

RELATIVE PERCENTAGES OF CLAY MINERALS IN REPLICATE SLIDE MOUNTS* (mounting variance) FROM DISPERSIONS OF SINGLE 50-GRAM PORTIONS OF 14 SEDIMENT GRABS, FROM 12 STATIONS IN THIS STUDY (SL877)

		Clay Mineral			
Station	Slide	Expandable Minerals	Illite	Kaolinite	Chlorite
1a	1	5.5	66.8	9.7	18.0
	2	7.6	63.1	9.4	19.9
	3	7.0	64.9	8.9	19.2
5a	1**	5.3	65.4	10.7	18.6
	2	8.2	61.2	9.3	21.3
	3	5.8	62.8	10.6	20.8
8	1	2.0	64.4	12.2	21.4
	2	2.3	68.2	10.1	19.4
	3	2.7	66.0	10.3	21.0
12	1	7.2	63.1	9.5	20.2
	2	8.6	62.4	10.0	19.0
	3	9.4	60.4	10.1	20.1
17	1	2.0	65.3	13.9	18.8
	2	2.8	65.3	11.7	20.2
	3	2.2	65.2	11.5	21.1
21b***	1**	14.0	55.7	8.8	21.5
	2	17.1	53.0	9.9	20.0
21b†	5	11.1	58.6	10.1	20.2
	6	11.4	57.7	10.5	20.4
	7	13.0	56.0	10.5	20.5
21d	1**	3.7	67.1	11.8	17.4
	2**	5.5	68.5	10.5	15.5
22	1**	8.7	60.3	10.5	20.5
	2	9.5	59.8	10.7	20.0
	3	10.8	58.2	11.0	20.0
25b	1	11.3	59.8	10.0	18.9
	2**	11.5	58.5	11.4	18.6
	3	11.1	58.0	9.5	21.4

APPENDIX IV-C

(Continued)

Station	Slide	Clay Mineral			
		Expandable Minerals	Illite	Kaolinite	Chlorite
26	1	12.8	53.6	12.1	21.5
	2	12.4	54.9	11.1	21.6
	3	11.6	56.2	11.5	20.7
	4	12.5	55.8	11.3	20.4
	5	12.1	56.0	11.9	20.0
	6**	13.3	56.6	9.4	20.7
27	1	13.8	52.9	11.7	21.6
	2	12.3	55.7	9.8	22.2
	3	11.6	57.4	10.2	20.8
32b	1**	3.7	70.3	7.5	18.5
	2**	5.3	65.8	8.7	20.2
32d	1**	10.3	56.6	10.2	22.9
	2	9.2	56.4	9.7	24.7
36	1	10.4	54.1	12.2	23.3
	2**	8.8	59.0	9.7	22.5
	3**	11.1	59.0	9.9	20.0

* Less than 2μm fraction.
** Average values from Appendix IV-D.
*** Dispersion 1 in Appendix IV-B.
† Dispersion 4 in Appendix IV-B.

APPENDIX IV-D

RELATIVE PERCENTAGES OF CLAY MINERALS, DETERMINED FROM REPLICATE INSTRUMENT SCANS AND CALCULATIONS ON 14 SEPARATE SLIDE MOUNTS, FOR TEN SEDIMENT GRABS OF EIGHT STATIONS IN THIS STUDY (SL877)

Station /Slide	Analysis & Calculation	Clay Mineral			
		Expandable Minerals	Illite	Kaolinite	Chlorite
1c/1	1	10.9	59.4	8.9	20.8
	2	8.8	58.8	9.7	22.7
5a/1	1	5.1	65.5	11.3	18.1
	2	5.5	65.4	10.0	19.1
21b/1	1	13.4	56.1	8.9	21.6
	2	14.6	55.2	8.8	21.4
21b/3*	1	10.7	59.5	9.6	20.2
	2	11.4	58.4	11.0	19.2
21d/1	1	4.2	67.6	10.9	17.3
	2	3.2	66.7	12.6	17.5
21d/2	1	5.4	68.8	10.4	15.4
	2	5.5	68.3	10.5	15.7
22 /1	1	9.6	60.2	10.1	20.1
	2	7.8	60.3	10.9	21.0
25b/2	1	12.8	57.8	11.2	18.2
	2	10.6	57.7	12.0	19.7
	3	11.1	60.0	11.1	17.8
26 /6	1	14.6	56.2	9.3	19.9
	2	12.9	56.7	9.7	20.7
	3	11.5	57.2	9.5	21.8
	4	14.3	56.1	9.0	20.6
32b/1	1	3.8	69.9	7.4	18.9
	2	3.6	70.7	7.6	18.1
32b/2	1	5.3	66.7	8.5	19.5
	2	5.3	65.0	9.0	20.7

APPENDIX IV-D

(Continued)

Station /Slide	Analysis & Calculation	Clay Mineral			
		Expandable Minerals	Illite	Kaolinite	Chlorite
32d/1	1	10.6	56.0	11.0	22.4
	2	10.1	57.3	9.3	23.3
36 /2	1	8.8	60.1	9.4	21.7
	2	8.8	57.9	10.1	23.2
36 /3	1	10.9	58.8	10.1	20.2
	2	12.2	59.2	9.5	19.1
	3	10.3	59.1	10.2	20.4

* Dispersion 2 in Appendix IV-B.

APPENDIX V

RESULTS OF REPLICATE ANALYSES FOR TOTAL CARBON AND CARBONATE, AND CALCULATIONS OF ORGANIC CARBON, IN SIMPSON LAGOON SEDIMENTS, AND CALCIUM CARBONATE AND DEXTROSE STANDARDS

APPENDIX V-A

WEIGHT PERCENTS OF CARBON CONTENTS IN REPLICATE SEDIMENT GRABS (intra-station variance) RETRIEVED FROM FIVE STATIONS IN THIS STUDY (SL877)

Station	Total Carbon*	Carbonate**	Organic Carbon***
1a	1.01	3.77	0.26
1b	0.62	1.78	0.26
1c	0.78	2.20	0.34
1d	0.86	2.10	0.44
1e	0.97	4.05	0.16
5a	2.63	10.27	0.58
5b	2.76	9.90	0.78
5c	3.34	10.90	1.20
5d	3.10	10.53	0.99
21a	3.40	9.54	1.5
21b	3.40	10.30	1.3
21c	3.10	9.96	1.1
21d	3.27	8.95	1.5
25a	2.69	4.79	1.7
25b	2.22	4.16	1.4
25c	2.94	4.90	2.0
32a	3.88	12.60	1.4
32b	4.50	13.54	1.8
32c	3.95	11.32	1.7
32d	3.41	10.64	1.3

* Average of replicate analyses from Appendix V-B, where applicable.

** Average of replicate analyses from Appendix V-D, where applicable.

***Calculated from: total carbon minus carbonate carbon, where the latter amount is obtained by dividing carbonate by five.

APPENDIX V-B

TOTAL CARBON CONTENTS (weight percent), IN REPLICATE 0.2-GRAM PORTIONS (intra-subsample variance) OF 30-GRAM SAMPLE SPLITS FROM 23 SEDIMENT GRABS, FOR 19 STATIONS IN THIS STUDY (SL877)

	TOTAL CARBON Day* of Subsample Analysis			
Station	1	2	3	4
1b		0.604		0.640
1d	0.797	0.880		0.862
2		0.429		
		0.419		
		0.419		
4	3.26		3.55	3.60
5b		2.76		2.77
5c		3.31		3.36
6	4.44			4.45
7	1.40			1.43
9	4.26	3.77		4.29
11		2.54		2.86
12		3.64		
		3.63		
		3.58		
15	0.839		0.818	
17		2.08		
		2.05		
		2.04		
18	4.41	3.36		4.09
19	1.63		1.53	
20	0.727			0.757
21b	3.43		3.36	
27	3.34		3.10	3.27
32b		4.49		4.44
		4.69		
32c		3.92		3.97
32d		3.35		3.48
37		2.80		
		2.87		
		2.73		
UG-1	5.36		5.10	

* Separate instrument calibrations on different days.

APPENDIX V-C

CALCULATED TOTAL CARBON CONTENTS (weight percent) FROM THE ANALYSIS OF TEN 0.01-GRAM PORTIONS OF A DEXTROSE STANDARD (40% carbon by weight) BY THE LECO INDUCTION FURNACE USED IN THIS STUDY

Total Carbon
38.17
38.80
36.94
40.32
39.04
37.32
37.02
39.66
37.40
40.20

$\bar{X}$	s	Range
38.5	1.3	37-40.3

APPENDIX V-D

TOTAL WEIGHT PERCENT OF CARBONATE ($CO_3^{=}$) IN REPLICATE SUBSAMPLES* (intra-subsample variance) FROM 23 SEDIMENT GRABS OF 17 STATIONS IN THIS STUDY (SL877)

	$CO_3^{=}$ In Each Subsample			
Station	1	2	3	4**
1b	1.83			1.73
1c	2.25	2.15		
1d	2.00			2.19
1e	4.04	4.10		
4	8.58			8.91
5b	9.58			10.20
5c	10.37			11.50
6	9.00	9.22		10.20
7	5.78			6.31
9	9.42			9.53
11	10.73			11.37
14	7.29	7.71	8.51	
18	3.92			4.35
20	1.89			1.88
22	10.75	10.72		
25b	4.12	4.28		4.09
27	10.37			10.43
28	11.14	11.60		
29	11.21	9.98	10.21	
32b	13.22	13.51		13.89
32c	11.31			11.33
32d	11.53	10.21		10.17
33	7.77	7.15	8.15	

* 0.2-gram portions from 30-gram sample splits.
** Analysis performed by different technician.

APPENDIX V-E

CALCULATED $CaCO_3$ CONTENTS (weight percent) FROM THE ANALYSIS OF 21 0.02-GRAM PORTIONS OF A 100% $CaCO_3$ STANDARD BY THE MANOMETRIC METHOD* USED IN THIS STUDY

$CaCO_3$ Content
103.5
113.8
99.7
112.3
98.2
115.8
108.4
98.2
90.5
101.8
108.3
99.7
100.9
112.5
100.0
100.0
94.9
98.3
95.8
99.7
101.0

$\bar{X}$	s	Range
103	6.8	91-116

* Hulsemann, 1966.

APPENDIX VI

RESULTS OF REPLICATE ANALYSES FOR HEAVY METALS RELEASED FROM SIMPSON LAGOON SEDIMENTS AND STANDARD U.S. GEOLOGICAL SURVEY ROCK POWDERS, BY DIGESTION WITH HYDROFLUORIC-NITRIC ACID

APPENDIX VI-A

CONCENTRATIONS (µg/g, except Fe which is in 10^4 µg/g) OF HEAVY METALS IN REPLICATE SEDIMENT GRABS* (intra-station variance) TAKEN FROM FIVE STATIONS IN THIS STUDY (SL877), RELEASED BY DIGESTION WITH HYDROFLUORIC-NITRIC ACID (HFNA)

Station	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1a**	42.7	23	118	1.13	4.3	10.6	8.0	34.0
1b	35.6	19	114	1.02	3.1	8.1	8.3	***
1c	40.9	21	155	1.10	4.1	10.6	5.4	***
1d**	41.9	21	126	1.08	4.4	9.2	6.5	45.4
1e	41.9	24	139	1.23	4.4	12.8	7.1	44.9
5a	68.6	39	273	1.82	6.2	21.8	14.3	77.5
5b	61.5	38	235	1.79	5.8	20.8	23.1	72.7
5c	69.2	46	344	2.08	8.0	22.6	15.8	81.9
5d	69.6	46	316	2.17	8.6	22.8	21.4	79.8
21a**	100.1	66	370	2.81	9.6	30.5	22.4	100.5
21b	95.7	66	344	2.76	9.8	29.3	19.2	***
21c	85.9	61	257	2.46	8.5	29.2	17.2	103.2
21d	91.3	65	324	2.71	9.6	29.0	13.2	90.9
25a**	57.0	37	188	1.72	6.7	17.0	11.5	63.6
25b	56.6	33	165	1.59	6.0	15.4	15.7	85.5
25c	65.0	39	199	1.83	8.4	20.0	16.8	74.9
32a	97.2	52	299	2.25	8.4	27.1	16.0	93.0
32b	108.8	63	366	2.51	9.3	31.6	19.6	103.2
32c	96.6	56	302	2.39	9.3	29.3	22.4	98.0
32d	83.0	48	273	2.12	7.6	27.5	18.8	89.8

* Based on digestion of 0.5-gram subsamples.
** Average values from Appendix VI-B.
*** Extreme value not included because of probable contamination.

APPENDIX VI-B

CONCENTRATIONS (μg/g, except Fe which is in 10^4 μg/g) OF HEAVY METALS IN REPLICATE SUBSAMPLES* (intra-subsample variance) FROM TEN SEDIMENT GRABS OF NINE STATIONS IN THIS STUDY (SL877), RELEASED BY DIGESTION WITH HYDROFLUORIC-NITRIC ACID (HFNA)

Station	Digest	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1a	1	44.8	23.8	120	1.13	4.6	10.4	6.1	34.8
	2	43.4	23.3	120	1.13	3.8	11.3	8.1	32.1
	3	39.9	22.4	115	1.14	4.4	10.0	9.7	34.9
1d	1	41.9	21	129	1.07	4.4	10.6	4.6	58.8
	2	-	21	123	1.08	-	7.7	8.3	32.0
6	1	72	56.0	369	2.30	-	27.7	22.5	83.6
	2	78	52.4	-	2.30	7.9	27.3	-	-
7	1	42.2	25	150	1.22	5.8	13.8	10.6	39.0
	2	47.8	27	151	1.21	5.3	12.9	10.9	26.7
15	1	61.2	37	226	1.68	8.6	21.4	15.7	63.1
	2	-	39	227	1.66	9.4	19.3	14.0	58.8
18	1	77.0	58	165	1.96	9.6	26.5	21.3	74.2
	2	72.0	53	160	1.79	8.2	25.8	18.8	83.6
	3	68.5	55	163	1.99	8.9	27.9	19.9	77.0
	4	77.2	50	163	1.96	8.2	27.2	-	-
21a	1	99.5	65.6	368	2.78	9.8	30.3	26.0	93
	2	100.4	66.2	371	2.84	9.6	31.2	16.9	105
	3	100.3	65.9	372	2.81	9.5	30.0	24.4	103
25a	1	56.3	36	188	1.72	6.8	17.6	12.1	64.2
	2	57.8	38	188	1.73	6.6	16.4	11.0	63.1
27	1	85	58.0	295	2.49	9.2	30.2	15.0	78.9
	2	82	55.4	-	2.44	8.4	28.2	-	-
35	1	86	57	253	2.05	6.8	25.8	17.5	83.6
	2	86	53	244	2.03	7.4	25.2	17.5	88.2
	3	84	53	244	2.09	7.9	25.6	20.4	83.4

* Digests of 0.5-gram portions of 30-gram sample splits.

APPENDIX VI-C

CONCENTRATIONS (μg/g, except Fe which is in 10^4 μg/g) OF HEAVY METALS IN SUBSAMPLES OF THIS STUDY (SL877), DETERMINED FROM REPLICATE ASPIRATIONS* (Flame Atomic Absorption) OF SINGLE HYDROFLUORIC-NITRIC ACID DIGEST (HFNA) SOLUTIONS**

Aspiration	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1	78	34	547	2.16	3.7	28.2	6.1	97
2	79	32	557	2.15	4.0	26.9	6.1	97
3						27.4		
4						27.6		
1	92	58	507	1.86	4.8	9.5	19.9	84
2	93	56	516	1.82	4.5	10.5	19.9	84
1	107	51		1.30	3.2	27		34
2	107	48		1.26	3.2	25		39
1	89	22.1		2.31	4.3	26		78
2	87	22.6		2.30	4.8	25		78
3				2.29				
1	60	45.7		2.03	8.8	26		78
2	66	45.2		2.02	7.6	25		83
3					8.2			
1		36.2		0.96	9.8	28.2		88
2		36.7		0.96	10.0	27.6		83
1		50.8		1.06		25.9		58
2		52.8		1.06		25.2		63
3		51.8						
1		62.4		1.03		26.9		73
2		62.9		1.04		27.4		68
1		55.4		1.01		21.2		82
2		55.9		1.00		20.4		82
1		47.5				29.4		92
2		48.0				28.8		97
1		56						84
2		58						84
1								88
2								83

* Replicates run during the same instrument calibration.
** The same digest was not necessarily run for each metal.

APPENDIX VI-D

CONCENTRATIONS (μg/g, except for Fe which is in 10^4 μg/g) OF HEAVY METALS IN STANDARD U.S. GEOLOGICAL SURVEY AGV-1 and BCR-1 ROCK POWDERS, DETERMINED IN THIS STUDY AND REPORTED BY FLANAGAN (1969, 1973)

U.S.G.S. Standard Sample	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
AGV-1								
This Study-1		9.6		4.67	15.0	13.0	59.2	99
2	127	12.8	767	4.79	14.6	14.5		
Reported Literature Values:								
Average*	121	12.9	728	4.76	15.5	17.8	63.7	112
Average**	125	12.2	763	4.73	14.1	18.5	59.7	84
Range*	70-171	8-45	640-870	4.26-5.21	10-30	11-27	52-83	63-304
BCR-1								
This Study-1		10.7			33.7	5.9	17.5	136
2	500	14.6	1425	8.92	35.7	6.4		
Reported Literature Values:								
Average*	384	16.3	1350	9.44	35.5	15.0	22.4	132
Average**	399	17.6	1406	9.37	38	15.8	18.4	120
Range*	120-700	8-45	1040-1600	9.02-9.97	29-60	8-30	7-33	94-278

* Flanagan, 1969.
** Flanagan, 1973.

APPENDIX VII

RESULTS OF REPLICATE ANALYSES FOR HEAVY METALS RELEASED FROM SIMPSON LAGOON SEDIMENTS BY THE HYDROXYLAMINE HYDROCHLORIDE-ACETIC ACID REAGENT

APPENDIX VII-A

CONCENTRATIONS (μg/g) OF HEAVY METALS IN REPLICATE SEDIMENT GRABS* (intra-station variance) TAKEN FROM FIVE STATIONS OF SIMPSON LAGOON, EXTRACTED BY THE HYDROXYLAMINE HYDROCHLORIDE-ACETIC ACID REAGENT (HHAA)**

Station	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1a***	1.94	0.22	48.0	1120	0.72	0.89	1.92	5.4
1b***	1.79	0.27	40.7	940	0.44	0.67	1.74	4.0
1c	2.36	0.65	89.9	1420	0.88	1.20	1.62	4.8
1d†	1.92	0.39	43.6	780	0.55	0.72	1.23	4.4
1e	2.31	0.90	54.0	1250	0.85	1.52	1.80	5.5
5a	3.6	1.5	155	2280	1.90	3.28	2.36	11.4
5b†	3.3	1.4	128	2000	1.80	2.65	8.9‡	18.5‡
5c	4.1	1.4	208	2710	2.55	3.74	2.92	13.4
5d	3.6	1.0	169	2250	1.88	3.05	2.94	12.5
21a	6.5	1.2	193	3550	3.3	4.65	3.52	18.7
21b†	6.0	1.4	165	3010	3.4	4.78	2.96	17.0
21c***	6.4	1.6	108	2360	2.5	3.76	3.72	17.1
21d***	5.3	1.6	135	2800	2.5	3.68	3.61	15.4
25a	3.1	0.94	77.6	1720	1.82	2.74	2.28	10.5
25b	2.5	0.95	64.3	1490	2.15	2.73	2.34	9.2
25c***	3.1	1.2	77.0	1820	2.00	3.42	2.35	11.2
32a†	5.0	1.50	198	2690	2.7	6.05	3.11	22.6
32b	4.5	1.73	203	2630	2.8	5.93	3.08	20.9
32c	4.2	1.40	148	2340	2.5	4.72	2.88	18.0
32d†	4.0	1.05	146	2230	2.2	4.12	3.19	16.5

* Extraction was performed on one-gram portions of the 50-gram samples taken from each grab.
** Chester and Hughes, 1967.
*** Values are averages from Appendix VII-B.
† Values are averages from Appendix VII-C.
‡ Extreme value omitted from analysis of variance.

APPENDIX VII-B

CONCENTRATIONS (μg/g) OF HEAVY METALS IN REPLICATE SUBSAMPLES* (intra-subsample variance) FROM SEVEN SEDIMENT GRABS OF FIVE STATIONS IN THIS STUDY (SL877), EXTRACTED BY THE HYDROXYLAMINE HYDROCHLORIDE-ACETIC ACID REAGENT (HHAA)**

Station	Extract	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1a	1	1.95	0.23	48.8	1125	0.78	0.87	2.00	5.50
	2	2.05	0.32	51.3	1199	0.67	1.04	1.92	5.61
	3***	1.81	0.12	43.8	1030	0.70	0.75	1.85	5.13
1b	1	1.60	0.21	41.1	978	0.45	0.63	1.79	3.74
	2	1.81	0.19	37.3	882	0.38	0.64	1.52	3.74
	3	1.96	0.40	43.7	967	0.48	0.75	1.91	4.50
4	1	7.2		345		4.0		4.2	
	2	7.8		382	4600			5.1	21.3
	3***	6.5	1.9	376	4650	3.4	5.27	5.7	21.3
	4	7.5	2.0	360	4700	3.5	5.36	4.1	20.6
14	1			222	2700				
	2							3.50	
	3	4.3						1.70	
	4***	4.2	1.3	205	2768	2.7	3.79	2.88	12.2
	5	4.0	1.2	211	2626	2.5	3.75	2.17	12.5
21c	1	6.2	1.4	103	2180	2.2	3.25	3.46	16.7
	2	6.6	1.8	113	2532	2.6	4.27	3.97	17.5
21d	1***	5.6	1.7	138	2892	2.6	3.49	3.56	16.0
	2***	5.1	1.5	130	2541	2.4	3.42	3.42	14.5
	3	5.0	1.6	129	2432	2.4	3.04	3.47	14.7
	4	5.4	1.6	145	3351	2.5	4.77	3.97	16.5
25c	1***	3.0	1.0	74.4	1757	1.8	3.21	2.17	10.4
	2***	3.0	1.3	75.4	1757	2.1	3.51	2.24	11.1
	3	3.4	1.2	82.2	1934	2.0	3.54	2.64	12.2

* Extractions performed on one-gram portions of 50-gram sample splits taken from each sediment grab.

** Chester and Hughes, 1967.

*** Average values from Appendix VII-C were used.

APPENDIX VII-C

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS FROM REPLICATE ATOMIC ABSORPTION ANALYSES* AND CALCULATIONS (calibration variance) ON 13 SEPARATE HYDROXYLAMINE HYDROCHLORIDE-ACETIC ACID EXTRACTS OF SUBSAMPLES FROM 8 STATIONS IN THIS STUDY (SL877)

Station /Extract	Anal. & Calc.	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1a/3	1	1.78	0.10			0.62	0.74		
	2	1.84	0.14			0.77	0.75		
1d/2	1		0.40	43.6	781		0.62	1.12	4.5
	2		0.13	44.3	786		0.82	1.33	4.5
	3								4.1
4/3	1			383	4651			5.66	20.7
	2			368	4650			5.74	21.8
	3								21.5
5b	1	3.4	1.5	129	1998	1.8	2.63	9.10	18.9
	2	3.1	1.1	126	2003	1.7	2.67	8.76	18.7
	3								17.9
12	1			136	2464		3.53	3.15	16.5
	2			133	2549		3.98	2.77	15.7
14/4	1			203	2814			3.00	12.2
	2			207	2722			2.77	12.2
21b	1			165	3007			3.09	16.7
	2			168	2971			2.82	17.3
21d/1	1								15.4
	2								16.5
21d/2	1	5.1	1.5	130	2475	2.5	3.46	3.51	14.7
	2	5.0	1.4	131	2607	2.3	3.38	3.33	14.4
25c/1	1								10.2
	2								10.6
25c/2	1			74.5	1760		3.38	2.17	10.7
	2			76.2	1754		3.64	2.31	11.5
32a	1	4.5	1.5			2.5	5.85		
	2	5.5	1.4			2.9	6.24		
32d	1	3.6	1.05			2.1	4.09		
	2	4.3	1.08			2.3	4.16		

* V, Cr, Co, Ni: graphite furnace.

APPENDIX VII-D

CONCENTRATIONS (μg/g) OF HEAVY METALS IN SUBSAMPLES OF THIS STUDY (SL877), DETERMINED FROM REPLICATE ASPIRATIONS* ON SINGLE HYDROXYLAMINE HYDROCHLORIDE-ACETIC ACID EXTRACTS**

Aspiration***	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1	2.1	0.30	192	948	0.63	0.54	1.12	5.5
2	1.7	0.35	192	924	0.59	0.86	1.10	5.5
3	1.9	0.38			0.62	0.62		
1	1.5	0.18	360	2210	0.34	2.95	1.84	5.5
2	1.8	0.13	358	2250	0.37	2.80	1.74	5.8
3	2.0	0.13		2300	0.44	2.96		
4	1.9							
1	2.1	0.40	340	2440	3.29	0.75	2.85	5.0
2	2.8	0.47	337	2415	3.49	0.93	2.95	5.3
3	3.7	0.45			3.27	0.93		
4	3.0				3.32			
5					3.26			
6					3.55			
1	3.27	1.7	139	2790	1.81	3.72	3.26	4.7
2	3.57	1.8	138	2830	1.80	4.04	3.41	5.3
3	3.30	1.8		2880	1.86	3.62	3.36	
1	1.5	1.45		2640	2.7	3.07	2.75	17.0
2	0.9	1.65		2640	2.5	3.31	2.85	16.4
3	0.8	1.32			2.8	3.37		
4	1.2	1.45			2.8			
5					2.7			
1	2.0	1.10		2265	3.3	4.40	1.68	14.5
2	1.6	0.99		2290	3.3	4.59	1.63	15.2
3	1.7	1.01		2310	3.3	4.48		15.4
1	5.5	0.60		2380	2.4	2.81		10.5
2	6.0	0.60		2430	2.5	3.12		10.7
3	6.2	0.52		2480	2.6	3.01		
1	6.6	0.89		2635	2.8	4.3		10.7
2	6.1	0.71		2600	2.3	4.6		10.7
3	6.0	0.73			2.2	4.5		
1	6.0	1.24			2.4	5.22		12.0
2	5.5	1.37			2.5	5.33		12.3
3	5.9	1.19			2.6	5.56		
4						5.31		

APPENDIX VII-D

(Continued)

Aspiration***	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1	4.65	0.32			2.5	5.9		16.7
2	4.56	0.41			2.5	5.6		16.2
3	4.80	0.46			2.6	5.3		
4						5.5		
1	3.9	1.18			1.9	6.14		2.5
2	4.4	1.05			1.8	6.16		2.2
3	4.8	1.05			1.9	6.08		
4	3.9							
1	1.8	1.42			2.60	1.31		15.8
2	1.5	1.37			2.75	1.36		15.5
3	2.5	1.37			2.66	1.15		
4		1.44			2.59			
5					2.65			
1	2.97	2.16			3.45	3.9		16.2
2	3.05	1.82			3.35	3.9		15.9
3	3.08	2.06			3.38	4.1		
4		2.04						
1	4.7	1.53			2.35	1.43		22.2
2	5.1	1.55			2.27	1.43		22.4
3	5.4	1.46			2.34	1.59		22.5
4	5.1							
1	5.35	2.45			2.80	4.62		14.9
2	4.97	2.10			2.81	4.62		14.8
3	5.13	2.19			3.01	4.62		
4		2.40						
1		1.27			2.16	4.15		17.5
2		1.13			2.16	4.20		17.7
3		1.19			2.11	4.34		17.9
4								17.7
1		0.36			2.51	0.76		3.16
2		0.42			2.70	0.63		3.24
3		0.53			2.64	0.67		3.20
4					2.55	0.65		
5						0.69		
1		1.52			2.84	4.38		
2		1.58			2.99	4.75		
3		1.36			3.09	4.94		
4					3.14	4.70		
5					2.93			

APPENDIX VII-D

(Continued)

Aspiration***	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1		1.44			0.93	4.1		
2		1.45			0.84	4.2		
3		1.31			0.84	4.4		
1		1.40			2.53	4.04		
2		1.39			2.54	4.13		
3		1.38			2.53	4.16		
4					2.50			

* V, Cr, Co, Ni: graphite furnace.
** Within any aspiration group, results from different extracts may be listed for different metals.
*** Replicates run during same analysis.

APPENDIX VIII

RESULTS FOR HEAVY METALS REMOVED BY THE FIRST SEVEN STEPS OF THE EIGHT-STEP EXTRACTION SCHEME, AND THE ATOMIC ABSORPTION TECHNIQUES USED TO ANALYZE EACH METAL IN EXTRACTS FROM ALL EIGHT STEPS, FOR REPLICATE SEQUENCES PERFORMED ON SUBSAMPLES FROM SIX STATIONS IN THIS STUDY

APPENDIX VIII-A

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED** BY STEP ONE*** IN REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME USED IN THIS STUDY

Station	Sequence	V	Cr	Mn	Fe	Co	Ni	Cu
2	2	0.13	0.027	10.2	3.0	0.025	0.093	0.29
	4	0.12	0.009	9.4	0.1	0.018	0.023	0.18
	5	0.15	0.021	8.5	1.7	0.013	0.057	0.21
13	2	0.25	0.068	36	9.4	0.061	0.25	0.69
	4	0.11	0.023	32	18.5	0.027	0.18	0.61
	5	0.26	0.092	42	22.0	0.075	0.25	0.94
23	2	0.18	0.045	26.0	4.8	0.039	0.24	0.61
	4	0.74	0.330	11.6	41.0	0.050	0.25	1.00
	5			28.0	9.5			
	6	0.17	0.036	24.5	10.7	0.023	0.19	0.54
UG- 1	2	0.40	0.040	17.4	18	0.036	0.21	0.49
	4	0.29	0.061	16.6	32	0.025	0.25	0.45
	5			16.3	28			

* Results for zinc prevented by general contamination problem.
** Extractions performed on two-gram portions from 50-gram splits of sediment grabs from four stations in this study (SL877).
*** See Table 5 for explanation.

APPENDIX VIII-B

SEDIMENT CONCENTRATIONS (µg/g) OF HEAVY METALS REMOVED* BY STEP TWO** IN REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME USED IN THIS STUDY

Station	Sequence	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
2	1	0.23	0.05	10	35	0.07	0.0	0.00	2.4
	2	0.23	0.29	15	90	0.15	0.7	0.75	1.5
	3	0.12	0.17	40	50	0.44	1.0	0.40	5.2
	4	0.00	0.00	6	0	0.00	0.0	0.00	0.0
	5	0.00	0.23	34	23	0.25	1.1	2.3	6.0
	6	0.15	0.00	20	30	0.08	0.0	0.00	0.0
13	1	0.65	0.37	49	90	0.28	0.9	C***	7.0
	2	1.3	3.1†	51	300	0.17	2.3	0.0	8.7
	3	0.28	0.45	80	3	0.07	1.5	1.7	12.0
	4	0.64	0.48	80	150	0.45	3.2	4.1	12.5
	5	0.20	0.13	92	145	0.71	1.0	2.4	6.5
23	1	1.5	0.78	39	190	0.35	1.6	0.0	10
	2	1.0	1.4	35	280	0.38	2.8	1.7	11
	3	2.5	0.20	130	480	1.4	2.5	C***	21
	4	1.3	0.39	85	260	0.57	2.7	3.7	10
	5	1.5	0.50	86	285	0.93	1.8	1.5	16
	6	1.5	0.38	55	115	0.33	0.4	0.0	0
28	4	0.92	0.38	27	185	0.50	6.3	5.6	11.5
	6	1.6	0.23	30	420	0.42	1.0	2.2	3.5
UG- 1	1	2.0	1.1	32	225	0.40	3.5	0.04	15.0
	2	0.40	0.21	20	185	0.26	5.8	4.7	8.5
	3	0.0	0.03	42	115	0.52	1.5	0.65	8.1
	4	2.2	0.38	45	320	0.58	6.0	4.0	16.5
	5	0.84	0.45	38	270	0.65	4.2	3.4	12.0

* Extractions performed on two-gram portions from 50-gram splits of sediment grabs from five stations in this study (SL877).

** See Table 5 for explanation.

*** Extreme value not included because of probable contamination.

† Extreme value omitted from analysis of variance.

APPENDIX VIII-C

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED** BY STEP THREE*** IN REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME USED IN THIS STUDY

Station	Sequence	V	Cr	Mn	Fe	Co	Ni	Cu
2	1	0.084	0.007	38	75	0.23	0.00	0.6
	2	0.012	0.004	32	230	0.29	0.00	1.2
	3	0.54	0.068	0	410	0.018	0.066	1.0
	4	0.30	0.030	3	450	0.092	0.10	2.2
	5	0.00	0.035	0	34	0.016	0.021	1.1
	6	0.16	0.025	32	87	0.28	0.006	0.5
13	2	0.36	0.051	120	750	0.66	0.006	5.2
	5	0.016	0.005	0	45	0.016	0.006	1.3
18	1	1.3	0.067	0.0	580	0.23	0.014	4.2
	3	1.3	0.10	0.0	2150	0.037	0.056	4.8
	4	0.28	0.003	0.0	480	0.007	0.00	0.2
	6	0.56	0.092	2.1	1065	0.71	0.15	9.5
23	1	0.60	0.016	36	170	0.092	0.00	2.5
	2	0.24	0.045	57	590	0.45	0.16	7.8
	3	0.65	0.27	2.0	870	0.086	0.065	5.1
	4	0.06	0.21	5.5	470	0.13	0.13	1.0
	5	0.034	0.26	0.0	72	0.047	0.014	1.1
	6	0.40	0.002	49	220	0.25	0.00	2.9
28	1	0.41	0.031	2.5	410	0.15	0.00	4.1
	3	1.9	0.16	0.0	1170	0.03	0.001	3.5
	4	0.0	0.002	10	740	0.13	0.00	4.5
	6	1.2	0.05	6.6	380	0.27	0.005	3.6
UG- 1	1	0.64	0.048	0.9	180	0.076	0.00	1.6
	2	0.057	0.037	17	280	0.21	0.16	2.9
	5	0.024	0.021	0.0	48	0.029	0.024	1.0

* Results for zinc prevented by general contamination problem.
** Extractions performed on two-gram portions from 50-gram splits of sediment grabs from six stations in this study (SL877).
***See Table 5 for explanation.

APPENDIX VIII-D

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS REMOVED* BY STEP FOUR** IN REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME USED IN THIS STUDY

Station	Sequence	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
2	1	0.15	0.008	3	57	0.052	0.19	0.19	1.0
	2	0.16	0.041	4	131	0.13	0.17	0.79	2.2
	3	0.12	0.026	6	94	0.07	0.22	0.23	0.8
	4	0.17	0.051	8	112	0.16	0.053	0.16	1.2
	5	0.08	0.004	10	106	0.10	0.083	0.096	1.0
	6	0.69	0.037	63	104	0.67	0.35	0.59	5.3
13	1	0.70	0.011	11	18	0.033	0.049	0.063	0.6
	2	0.20	0.22	10	300	0.37	0.73	0.56	1.8
	3	1.2	0.021	68	13	0.03	0.11	0.046	1.4
	4	0.24	0.013	52	25	0.055	0.034	0.21	0.9
	5	0.19	0.005	42	70	0.20	0.037	0.00	1.8
18	3	0.68	0.00	6	252	0.31	0.43	0.00	1.3
	4	0.87	0.36	4	431	0.39	1.4	0.32	3.2
	6	2.2	0.026	26	56	0.19	0.22	0.24	3.1
23	1	0.28	0.11	10	260	0.33	0.83	0.01	2.3
	2	0.62	0.025	10	48	0.15	0.24	0.00	5.4
	3	0.39	0.20	25	510	0.34	1.1	0.22	3.7
	4	0.32	0.042	25	79	0.14	0.057	0.23	3.2
	5	0.26	0.13	25	432	0.61	1.2	0.66	4.3
	6	0.29	0.011	27	53	0.24	0.14	0.14	1.5
28	3	0.90	0.00	9	180	0.24	0.58	0.00	1.5
	4	0.45	0.052	14	63	0.022	0.029	0.005	0.9
	6	4.1	0.050	33	8	0.018	0.048	0.46	0.7
UG- 1	1	0.37	0.082	6	108	0.12	0.21	0.02	1.1
	2	0.40	0.027	12	112	0.24	1.1	0.086	3.2
	3	0.83	0.11	19	288	0.42	1.5	0.13	5.0
	4	0.19	0.014	10	191	0.16	0.23	0.00	2.0
	5	0.15	0.003	11	134	0.047	0.060	0.00	1.6

* Extractions performed on two-gram portions from 50-gram splits of sediment grabs from six stations in this study (SL877).

** See Table 5 for explanation.

APPENDIX VIII-E

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS REMOVED* BY STEP FIVE** IN REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME USED IN THIS STUDY

Station	Sequence	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
2	1	2.8	0.20	2.0	1100	0.059	0.26	1.2	0.2
	2	1.5	0.14	0.1	1280	0.027	0.16	1.2	0.0
	3	0.5	0.08	8.8	725	0.44	0.60	1.3	3.1
	4	1.2	0.07	5.9	670	0.20	0.81	0.6	3.3
	5	1.0	0.17	8.8	1440	0.30	0.48	0.5	4.8
	6	2.5	0.31	4.3	1490	0.038	0.19	1.3	0.0
13	1	3.9	0.78	11	4260	0.54	2.1	5.7	2.2
	2	4.2	0.80	7.7	4180	0.15	1.2	2.0	0.8
	5	3.9	1.5	55	5365	1.8	2.7	5.2	17.5
18	3	3.7	1.1	10.8	3270	0.81	2.6	4.3	7.6
	4	4.9	0.19	7.0	3180	0.95	4.2	3.1	8.7
	6	6.1	1.4	2.6	6120	0.33	1.6	6.4	0.5
23	1	5.5	1.2	5.0	3800	0.23	2.0	1.5	1.0
	2	4.5	0.83	10.1	4320	0.48	1.5	3.8	1.5
	3	4.6	1.5	42	4270	3.0***	6.3***	4.7	12.5
	4	2.7	0.27	31	3445	1.1	2.9	5.6	13.8
	5	2.8	0.76	28	3865	1.0	2.3	6.3	10.5
	6	5.3	1.3	4.6	4700	0.21	1.1	6.6	0.5
28	3	2.4	0.69	30	3090	1.7	4.0	4.5	12.4
	4	6.0	0.60	25	3460	1.7	5.3	3.2	17.1
	6	6.0	1.5	13	5880	0.29	1.0	9.3	0.8
UG- 1	1	3.4	0.87	1.5	2220	0.09	1.5	1.4	0.5
	2	2.9	0.66	2.7	3700	0.37	1.0	2.1	1.1
	5	2.1	0.92	13	3040	0.71	2.9	5.0	11

* Extractions performed on two-gram portions from 50-gram splits of sediment grabs from six stations in this study (SL877).

** See Table 5 for explanation.

*** Extreme value omitted from analysis of variance.

APPENDIX VIII-F

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED** BY STEP SIX*** IN REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME USED IN THIS STUDY

Station	Sequence	V	Cr	Mn	Fe	Co	Ni	Cu
2	1	2.4	0.80	12	2056	0.8	0.8	0.4
	2	2.1	0.06	12	1985	0.6	0.7	0.7
	3	1.3	0.50	11	1860	0.9	1.0	0.2
	4	1.7	0.04	12	2265	0.9	0.5	0.3
	5	2.2	0.58	13	2575	0.8	0.9	0.1
	6	1.5	0.00	13	1790	0.8	0.6	0.0
13	1	4.2	1.7	44	3818	2.0	4.4	1.7
	2	3.4	0.54	36	4617	1.8	3.8	2.2
	3	3.9	1.7	41	3610	2.2	3.2	1.4
	5	3.3	0.42	37	4267	1.9	2.6	0.7
18	3	1.6	1.2	9	1280	1.8	1.8	0.7
	4	1.0	0.02	7	1700	0.8	1.0	0.5
	6	1.4	0.34	10	1530	2.2	2.0	0.9
23	1	3.2	1.8	42	5425	2.1	4.0	1.6
	2	2.8	0.86	38	4320	1.1	2.5	2.1
	3	3.0	1.9	38	5000	2.0	4.2	1.0
	4	2.7	0.06	36	4880	1.3	2.0	1.0
	5	2.5	0.53	35	4975	1.8	2.8	0.7
	6	3.0	0.50	41	4750	1.8	3.5	1.7
28	3	4.7	1.2	39	4895	2.1	3.9	1.4
	4	3.8	0.45	36	5085	1.9	2.3	1.3
	6	4.5	0.55	50	4775	2.1	4.4	0.9
UG- 1	1	4.2	2.1	24	3810	1.6	2.4	1.0
	2	2.9	0.32	29	3965	1.6	2.1	1.5
	3	5.0	3.0	26	3480	2.0	3.5	1.6
	5	3.4	0.34	24	3745	1.5	1.3	0.8

* Results for zinc prevented by general contamination problem.

** Extractions performed on two-gram portions from 50-gram splits of sediment grabs from six stations in this study (SL877).

*** See Table 5 for explanation.

APPENDIX VIII-G

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED** BY STEP SEVEN*** IN REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME USED IN THIS STUDY

Station	Sequence	V	Cr	Mn	Fe	Co	Ni	Cu
2	1	0.55	0.28	6	560	0.17	1.1	1.3
	2	1.1	0.55	5	740	0.33	1.1	0.7
	3	0.46	0.37	5	440	0.54	1.2	1.2
	4	1.0	0.30	4	395	0.30	0.79	1.1
	5	0.35	0.12	5	455	0.00	1.5	0.8
	6	0.75	0.25	6	425	0.45	0.64	1.1
13	1	5.0	3.5	50	4000	0.91	6.9	4.0
	2	2.8	3.2	28	4000	1.7	5.4	1.7
	3	5.6	4.6	39	2830	2.0	4.8	4.1
	5	4.5	4.3	29	3115	0.84	5.0	3.1
18	3	2.7	2.3	16	2070	1.4	3.6	2.3
	4	1.2	1.7	12	1700	0.70	2.8	1.2
	6	2.1	2.2	20	2470	1.8	4.2	6.5
23	1	2.8	2.9	20	2500	0.0	4.0	1.8
	2	2.5	2.3	25	3251	1.3	4.1	5.3
	3	1.8	2.8	11	1480	1.6	2.5	3.4
	4	1.5	2.2	17	2135	0.85	2.9	2.4
	5	2.0	2.3	12	1670	0.3	3.3	3.0
	6	1.7	1.8	23	2240	0.87	3.7	3.0
28	3	1.9	1.5	22	2360	1.0	3.6	4.1
	4	2.4	2.4	24	2565	1.1	3.8	2.4
	6	3.3	2.0	43	3265	1.0	4.8	4.5
UG- 1	1	2.7	3.3	17	1960	0.50	4.0	2.3
	2	1.8	1.8	22	2700	0.92	4.0	1.3
	3	2.6	2.7	11	1250	1.2	2.7	3.0
	5	2.0	2.6	13	1600	0.31	3.7	2.1

* Results for zinc prevented by general contamination problem.

** Extractions performed on two-gram portions from 50-gram splits of sediment grabs from six stations in this study (SL877).

*** See Table 5 for explanation.

APPENDIX VIII-H

METHODS OF ANALYSIS* FOR HEAVY METALS IN SOLUTIONS FROM EACH STEP** OF THE EIGHT-STEP EXTRACTION SCHEME

Step	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1	HGA	HGA	603	603	HGA	HGA	HGA	C***
2	HGA	HGA	603	603	HGA	HGA	HGA	603
3	HGA	HGA	603	603	HGA	HGA	603	C***
4	HGA	HGA	603	603	HGA	HGA	HGA	603
5	HGA	HGA	603	603	HGA	HGA	603	603
6	HGA	HGA	603	603	HGA	HGA	603	C***
7	HGA	HGA	603	603	HGA	603	603	C***
8	NA†	603	603	603	NA†	603	603	603

* HGA: HGA-500 graphite furnace; 603: Model 603 flame.
** See Table 5 for explanation.
*** Results prevented by general contamination problem.
† Not analyzed.

APPENDIX IX

RESULTS FOR THE SUMS OF HEAVY METALS REMOVED BY STEPS 1-4: (LEACHABLE FRACTIONS) AND STEPS 5-7: (IRON PHASES), FOR REPLICATE SEQUENCES OF THE EIGHT-STEP SCHEME PERFORMED ON SUBSAMPLES FROM SIX STATIONS IN THIS STUDY

APPENDIX IX-A

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED BY THE SUMS OF STEPS DESIGNATED "LEACHABLE FRACTIONS" AND "IRON PHASES", FOR REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME PERFORMED ON SUBSAMPLES OF THE SAND SL877-2

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				VANADIUM			
Leachable Fractions I	1	0.13	0.13	0.05	0.12	0.15	0.13
	2	0.23	0.23	0.12	0.00	0.00	0.15
	3	0.08	0.20	0.54	0.70	0.00	0.00
	4	0.15	0.16	0.12	0.17	0.08	0.69
	SUM	0.59	0.72	0.83	0.99	0.23	0.97
Iron Phases II	5	2.8	1.5	0.46	1.2	1.0	2.5
	6	2.4	2.1	1.3	1.7	2.2	1.5
	7	0.55	1.1	0.46	1.0	0.35	0.75
	SUM	5.8	4.7	2.2	3.9	3.6	4.8
				CHROMIUM			
Leachable Fractions I	1	0.020	0.03	0.090	0.01	0.021	
	2	0.052	0.29	0.17	0.00	0.23	
	3	0.007	0.01	0.068	0.12	0.035	
	4	0.007	0.04	0.026	0.05	0.004	
	SUM	0.086	0.37	0.35	0.18	0.29	
Iron Phases II	5	0.20	0.14	0.08	0.07	0.17	0.31
	6	0.80	0.06	0.50	0.7	0.58	0.00
	7	0.28	0.55	0.37	0.30	0.12	0.25
	SUM	1.3	0.75	0.95	1.1	0.87	0.56
				MANGANESE			
Leachable Fractions I	1	5	10	4	10	9	9
	2	10	15	40	6	34	20
	3	38	33	0	3	0	0
	4	3	4	6	8	10	63
	SUM	56	62	50	27	53	92
Iron Phases II	5	2	0.1	9	6	9	4
	6	12	12.4	11	12	13	12.8
	7	6	5.3	5	4	5	6.2
	SUM	20	18	25	22	27	23

APPENDIX IX-A

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				COBALT			
Leachable	1	0.01	0.03	0.01	0.02	0.013	0.02
Fractions	2	0.07	0.15	0.44	0.00	0.25	0.08
I	3	0.23	0.29	0.02	0.14	0.016	0.02
	4	0.05	0.13	0.07	0.16	0.10	0.67
	SUM	0.36	0.60	0.54	0.32	0.38	0.79
Iron	5	0.059	0.027	0.44	0.20	0.30	0.038
Phases	6	0.80	0.64	0.90	0.93	0.80	0.81
II	7	0.17	0.33	0.54	0.30	0.00	0.45
	SUM	1.0	1.0	1.9	1.4	1.1	1.3
				NICKEL			
Leachable	1	0.075	0.093	0.035	0.023	0.057	0.05
Fractions	2	0.00	0.66	1.0	0.00	1.1	0.00
I	3	0.00	0.00	0.066	0.10	0.021	0.03
	4	0.19	0.17	0.22	0.053	0.083	0.35
	SUM	0.26	0.92	1.32	0.18	1.26	0.43
Iron	5	0.26	0.16	0.60	0.81	0.48	0.19
Phases	6	0.80	0.72	1.0	0.53	0.9	0.60
II	7	1.1	1.1	1.2	0.80	1.5	0.64
	SUM	2.2	2.0	2.8	2.1	2.9	1.4
				COPPER			
Leachable	1	0.20	0.29	0.25	0.18	0.21	0.2
Fractions	2	0.00	0.75	0.40	0.00	2.3	0.0
I	3	0.56	1.2	1.0	2.2	1.1	1.0
	4	0.19	0.79	0.23	0.16	0.10	0.59
	SUM	0.95	3.0	1.9	2.5	3.7	1.8
Iron	5	1.2	1.2	1.3	1.6	0.5	1.3
Phases	6	0.35	0.7	0.2	0.3	0.15	0.0
II	7	1.3	0.73	1.2	1.1	0.8	1.1
	SUM	2.85	2.6	2.7	3.0	1.45	2.4

APPENDIX IX-A

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				IRON			
Leachable	1	5	3	4	0	2	2
Fractions	2	36	89	53	0	23	30
I	3	75	230	407	448	34	35
	4	57	131	94	112	106	104
	SUM	173	453	558	560	165	171
Iron	5	1100	1280	726	670	1440	1490
Phases	6	2060	1980	1860	2260	2580	1790
II	7	559	740	440	394	454	425
	SUM	3720	4000	3026	3324	4474	3705

* Zinc not included because of incomplete data.

APPENDIX IX-B

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED BY THE SUMS OF STEPS DESIGNATED "LEACHABLE FRACTIONS" AND "IRON PHASES", FOR REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME PERFORMED ON SUBSAMPLES OF THE SILT SL877-13

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				VANADIUM			
Leachable	1	0.25	0.25	0.35	0.11	0.26	
Fractions	2	0.65	1.3	0.28	0.64	0.20	
I	3	0.20	0.36	0.20	0.40	0.02	
	4	0.70	0.20	1.2	0.24	0.19	
	SUM	1.8	2.1	2.0	1.4	0.67	
Iron	5	3.9	4.2	4.0		3.9	
Phases	6	4.2	3.4	3.9		3.3	
II	7	5.0	2.8	5.6		4.5	
	SUM	13.1	10.4	13.5		11.7	
				CHROMIUM			
Leachable	1	0.06	0.068	0.07	0.023	0.092	
Fractions	2	0.37	3.1**	0.45	0.48	0.13	
I	3	0.02	0.051	0.10	0.10	0.005	
	4	0.01	0.22	0.02	0.013	0.005	
	SUM	0.46	3.4**	0.64	0.62	0.23	
Iron	5	0.78	0.80	1.0		1.5	
Phases	6	1.7	0.54	1.7		0.42	
II	7	3.5	3.2	4.6		4.3	
	SUM	6.0	4.5	7.3		6.2	
				MANGANESE			
Leachable	1	15	36			42	
Fractions	2	49	51			92	
I	3	100	121			0	
	4	11	10			42	
	SUM	175	218			176	
Iron	5	11	8	10		55	
Phases	6	43.5	36	41		37	
II	7	50	28	39		29	
	SUM	105.5	72	90		121	

APPENDIX IX-B

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				COBALT			
Leachable	1	0.018	0.06			0.075	
Fractions	2	0.28	0.17			0.71	
I	3	0.50	0.66			0.016	
	4	0.033	0.37			0.20	
	SUM	0.83	1.26			1.00	
Iron	5	0.54	0.15			1.8	
Phases	6	2.0	1.8			1.9	
II	7	0.91	1.7			0.84	
	SUM	3.45	3.65			4.54	
				NICKEL			
Leachable	1	0.095	0.25	0.12	0.18	0.25	
Fractions	2	0.93	2.3	1.5	3.2	1.0	
I	3	0.10	0.01	0.10	0.1	0.007	
	4	0.049	0.73	0.11	0.03	0.037	
	SUM	1.2	3.3	1.8	3.5	1.3	
Iron	5	2.1	1.3	2.0		2.7	
Phases	6	4.4	3.8	3.2		2.6	
II	7	6.9	5.4	4.8		5.0	
	SUM	13.4**	10.5	10.0		10.3	
				COPPER			
Leachable	1		0.70	1.0		0.9	
Fractions	2		0.00	1.7		2.4	
I	3		5.2	3.0		1.3	
	4		0.56	0.05		0.0	
	SUM		6.5	5.7		4.6	
Iron	5	5.7	2.0	4.0		5.2	
Phases	6	1.7	2.2	1.4		0.7	
II	7	4.0	1.7	4.1		3.1	
	SUM	11.4	5.9	9.5		9.0	

APPENDIX IX-B

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
			IRON				
Leachable	1		10			22	
Fractions	2		301			144	
I	3		750			43	
	4		300			71	
	SUM		1360			280	
Iron	5	4260	4180			5370	
Phases	6	3820	4620			4270	
II	7	4000	4000			3120	
	SUM	12080	12800			12760	

* Zinc not included due to incomplete data.
** Extreme value omitted from analysis of variance.

APPENDIX IX-C

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED BY STEPS FIVE THROUGH SEVEN**: "IRON PHASES", FOR REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME PERFORMED ON SUBSAMPLES OF THE PEATY, SILTY SAND SL877-18

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				VANADIUM			
Iron	5			3.7	4.9		6.1
Phases	6			1.6	1.0		1.4
II	7			2.7	2.2		2.1
	SUM			8.0	8.1		9.6
				CHROMIUM			
Iron	5			1.1	1.1		1.4
Phases	6			1.2	0.9		0.3
II	7			2.3	1.7		2.2
	SUM			4.6	3.7		3.9
				MANGANESE			
Iron	5			11	7		3
Phases	6			9	7		10
II	7			16	12		20
	SUM			36	26		33
				COBALT			
Iron	5			0.8	0.95		0.3
Phases	6			1.8	0.80		2.2
II	7			1.4	0.70		1.8
	SUM			4.0	2.45		4.3
				NICKEL			
Iron	5			2.6	4.2		1.6
Phases	6			1.8	1.0		2.0
II	7			3.6	2.8		4.2
	SUM			8.0	8.0		7.8

APPENDIX IX-C

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				COPPER			
Iron	5			4.3	3.1		6.4
Phases	6			0.7	0.5		0.9
II	7			2.3	1.2		6.5
	SUM			7.3	4.8		13.8
				IRON			
Iron	5			3270	3180		6120
Phases	6			1280	1700		1530
II	7			2070	1700		2470
	SUM			6620	6580		10120

* Zinc not included because of incomplete data.
** No sequences complete with steps 1-4.

APPENDIX IX-D

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED BY THE SUMS OF STEPS DESIGNATED "LEACHABLE FRACTIONS" AND "IRON PHASES", FOR REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME PERFORMED ON SUBSAMPLES OF THE SANDY SILT SL877-23

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				VANADIUM			
Leachable	1	0.31	0.18	0.50	0.74	0.40	0.17
Fractions	2	1.5	1.0	2.5	1.3	1.5	1.5
I	3	0.60	0.50	0.65	1.0	0.03	0.10
	4	0.28	0.62	0.39	0.32	0.26	0.29
	SUM	2.7	2.3	4.0	3.4	2.2	2.1
Iron	5	5.5	4.5	4.6	5.7	2.8	5.3
Phases	6	3.2	2.8	3.0	2.7	2.5	3.0
II	7	2.8	2.5	1.8	1.5	2.0	1.7
	SUM	11.5	9.8	9.4	9.9	7.3	10.0
				CHROMIUM			
Leachable	1	0.16	0.045	0.11	0.33	0.20	0.036
Fractions	2	0.78	1.4	0.20	0.39	0.50	0.38
I	3	0.02	0.10	0.27	0.80	0.26	0.25
	4	0.11	0.025	0.20	0.04	0.13	0.011
	SUM	1.1	1.6	0.78	1.6	1.1	0.68
Iron	5	1.2	0.83	1.5	1.8	0.76	1.3
Phases	6	1.8	0.86	1.9	1.5	0.53	0.50
II	7	2.9	2.3	2.8	2.2	2.3	1.8
	SUM	5.9	4.0	6.2	5.5	3.6	3.6
				MANGANESE			
Leachable	1	13	26	11	12	28	25
Fractions	2	39	35	127	85	86	55
I	3	36	57	2	7	0	0
	4	10	10	25	25	25	27
	SUM	98	128	165	129	139	107
Iron	5	5	10	42	32	28	5
Phases	6	42	38	38	36	35	41
II	7	20	25	11	17	13	23
	SUM	67	73	91	85	76	69

APPENDIX IX-D

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				COBALT			
Leachable Fractions I	1	0.018	0.04	0.027	0.05	0.03	0.02
	2	0.35	0.38	1.4	0.57	0.93	0.33
	3	0.092	0.45	0.086	0.49	0.05	0.05
	4	0.33	0.15	0.34	0.14	0.61	0.24
	SUM	0.79	1.0	1.85	1.25	1.6	0.64
Iron Phases II	5	0.23	0.48	3.0	1.1	1.0	0.21
	6	2.1	1.1	2.0	1.3	1.8	1.8
	7	0.0	1.3	1.6	0.85	0.30	0.87
	SUM	2.3	2.9	6.6**	3.25	3.1	2.9
				NICKEL			
Leachable Fractions I	1	0.10	0.24	0.11	0.25	0.18	
	2	1.6	2.8	2.5	2.7	1.8	
	3	0.0	0.16	0.07	0.83	0.01	
	4	0.83	0.24	1.1	0.06	1.2	
	SUM	2.5	3.4	3.8	3.8	3.2	
Iron Phases II	5	2.0	1.5	6.3	2.9	2.3	1.1
	6	4.0	2.5	4.2	2.0	2.8	3.5
	7	4.0	4.1	2.5	2.9	3.3	3.7
	SUM	10.0	8.1	13.0**	7.8	8.4	8.3
				COPPER			
Leachable Fractions I	1	1.0	0.6	0.84	1.0	0.8	0.54
	2	0.00	1.7	2.0	3.7	1.5	0.0
	3	2.5	7.8	5.1	2.6	1.1	1.0
	4	0.01	0.0	0.22	0.23	0.66	0.14
	SUM	3.5	10.1	8.2	7.5	4.1	1.7
Iron Phases II	5	1.5	3.8	4.7	5.6	6.3	6.6
	6	1.6	2.1	1.0	1.0	0.7	1.7
	7	1.8	5.3	3.4	2.4	3.0	3.0
	SUM	4.9	11.2	9.1	9.0	10.0	11.3

APPENDIX IX-D

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				IRON			
Leachable	1	16	5	14	41	10	
Fractions	2	192	278	482	259	285	
	3	169	590	872	613	72	
	4	260	48	510	79	432	
	SUM	637	921	1878**	992	799	
Iron	5	3800	4320	4270	3450	3860	4700
Phases	6	5430	4320	5000	4880	4980	4750
II	7	2500	3251	1480	2140	1670	2240
	SUM	11730	11891	10750	10470	10510	11690

* Zinc not included because of incomplete data.
** Extreme value omitted from analysis of variance.

APPENDIX IX-E

SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* REMOVED BY THE SUMS OF STEPS DESIGNATED "LEACHABLE FRACTIONS" AND "IRON PHASES", FOR REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME PERFORMED ON SUBSAMPLES OF THE CLAYEY SILT SL877-28

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				VANADIUM			
Leachable	1			0.3	0.34		0.3
Fractions	2			1.3	0.92		1.6
I	3			1.9	2.0		0.1
	4			0.9	0.45		4.1
	SUM			4.4	3.7		6.1
Iron	5			2.4	6.0		6.0
Phases	6			4.7	3.8		4.5
II	7			1.9	2.4		3.3
	SUM			9.0	12.2		13.8
				CHROMIUM			
Leachable	1			0.07	0.07		0.07
Fractions	2			0.30	0.38		0.23
I	3			0.16	0.2		0.03
	4			0.00	0.05		0.05
	SUM			0.53	0.70		0.38
Iron	5			0.69	0.60		1.5
Phases	6			1.2	0.45		0.55
II	7			1.5	2.4		2.0
	SUM			3.4	3.5		4.1
				MANGANESE			
Leachable	1			8	9		8
Fractions	2			30	27		30
I	3			0	11		0
	4			9	14		33
	SUM			47	61		71
Iron	5			30	26		13
Phases	6			39	36		50
II	7			22	23		43
	SUM			91	85		106

APPENDIX IX-E

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				COBALT			
Leachable	1			0.03	0.029		0.03
Fractions	2			0.45	0.50		0.42
I	3			0.03	0.53		0.10
	4			0.24	0.022		0.02
	SUM			0.75	1.08		0.57
Iron	5			1.7	1.7		0.29
Phases	6			2.1	1.9		2.1
II	7			1.0	1.1		1.0
	SUM			4.8	4.7		3.4
				NICKEL			
Leachable	1				0.24		0.20
Fractions	2				6.3		1.0
I	3				0.80		0.01
	4				0.03		0.05
	SUM				7.4		1.3
Iron	5			4.0	5.3		1.0
Phases	6			3.9	2.3		4.5
II	7			3.6	3.8		4.8
	SUM			11.5	11.4		10.3
				COPPER			
Leachable	1				0.72		0.7
Fractions	2				5.6		2.2
I	3				4.5		1.0
	4				0.01		0.5
	SUM				10.8		4.4
Iron	5			4.5	4.3		9.3
Phases	6			1.4	1.3		0.9
II	7			4.1	3.5		4.5
	SUM			10.0	9.1		14.7

APPENDIX IX-E

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				IRON			
Leachable	1			10	7		10
Fractions	2			300	185		420
I	3			1174	837		100
	4			180	63		8
	SUM			1660	1092		540
Iron	5			3090	3460		5880
Phases	6			4890	5090		4770
II	7			2360	2560		3270
	SUM			10340	11110		13920

* Zinc not included because of incomplete data.

APPENDIX IX-F

SEDIMENT CONCENTRATION (μg/g) OF HEAVY METALS* REMOVED BY THE SUMS OF STEPS DESIGNATED "LEACHABLE FRACTIONS" AND "IRON PHASES", FOR REPLICATE SEQUENCES OF THE EIGHT-STEP EXTRACTION SCHEME PERFORMED ON SUBSAMPLES OF THE PEATY, CLAYEY-SILT SAND UG-1

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				VANADIUM			
Leachable Fractions I	1	0.09	0.40	0.50	0.29	0.30	
	2	2.0	1.40	0.00	2.2	0.84	
	3	0.64	0.66	0.50	0.30	0.02	
	4	0.37	0.40	0.83	0.19	0.15	
	SUM	3.1	2.9	1.8	3.0	1.3	
Iron Phases II	5	3.4	2.9	2.5		2.1	
	6	4.2	3.9	5.0		3.4	
	7	2.7	1.8	2.6		2.0	
	SUM	10.3	8.6	10.1		7.5	
				CHROMIUM			
Leachable Fractions I	1	0.070	0.04			0.050	
	2	1.1	1.21			0.45	
	3	0.048	0.20			0.021	
	4	0.082	0.03			0.003	
	SUM	1.3	1.5			0.52	
Iron Phases II	5	0.87	0.66	0.8		0.92	
	6	2.1	1.32	3.0		0.34	
	7	3.3	2.8	2.7		2.6	
	SUM	6.3	4.8	6.5		3.9	
				MANGANESE			
Leachable Fractions I	1	9	17	10	17	16	
	2	32	20	42	46	38	
	3	20	21	1	1	0	
	4	6	12	19	10	11	
	SUM	67	70	72	74	65	
Iron Phases II	5	2	3	5		13	
	6	24	29	26		24	
	7	17	22	11		13	
	SUM	43	54	42		50	

APPENDIX IX-F

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				COBALT			
Leachable Fractions I	1	0.013	0.04	0.03	0.03	0.030	
	2	0.40	0.26	0.52	0.58	0.65	
	3	0.076	0.33	0.10	0.32	0.029	
	4	0.12	0.24	0.42	0.16	0.047	
	SUM	0.61	0.87	1.07	1.09	0.76	
Iron Phases II	5	0.09	0.37	0.4		0.71	
	6	1.6	1.6	2.0		1.5	
	7	0.50	0.92	1.2		0.31	
	SUM	2.2	2.9	3.6		2.5	
				NICKEL			
Leachable Fractions I	1	0.06	0.21	0.09	0.25	0.15	
	2	3.5	5.8	1.5	6.0	4.2	
	3	0.00	2.0	0.5	1.2	0.02	
	4	0.21	1.1	1.5	0.23	0.06	
	SUM	3.8	9.1	3.6	7.7	4.4	
Iron Phases II	5	1.5	1.0	2.0		2.9	
	6	2.4	2.1	3.5		1.3	
	7	4.0	4.0	2.7		3.7	
	SUM	7.9	7.1	8.2		7.9	
				COPPER			
Leachable Fractions I	1	0.50	0.49	0.69	0.45	0.50	
	2	0.04	4.7	0.65	4.0	3.4	
	3	1.6	2.9	2.0	1.8	0.9	
	4	0.02	0.09	0.13	0.0	0.0	
	SUM	2.2	8.2	3.5	6.3	4.8	
Iron Phases II	5	1.4	3.1	3.0		5.0	
	6	1.0	1.5	1.6		0.8	
	7	2.3	2.3	3.0		2.1	
	SUM	4.7	6.9	7.6		7.9	

APPENDIX IX-F

(Continued)

Extraction Group	Step	Sequence One	Two	Three	Four	Five	Six
				IRON			
Leachable	1	40	19	41	32	28	
Fractions	2	226	186	114	321	267	
I	3	178	297	200	163	48	
	4	108	112	288	191	134	
	SUM	552	614	643	707	477	
Iron	5	2220	3700	3000		3040	
Phases	6	3810	3960	3480		3740	
II	7	1960	2700	1250		1600	
	SUM	7990	10360**	7730		8380	

* Zinc not included because of incomplete data.
** Extreme value omitted from analysis of variance.

APPENDIX X

STANDARD DEVIATIONS, WITH RELEVANT STATISTICS, OF EACH SOURCE OF VARIATION STUDIED FOR ESTIMATES OF TEXTURAL, CLAY-MINERALOGICAL, AND CHEMICAL PARAMETERS OF SURFICIAL SEDIMENTS IN SIMPSON LAGOON

APPENDIX X-A

STANDARD DEVIATIONS (s), WITH RELEVANT STATISTICS, OF EACH SOURCE OF VARIATION STUDIED FOR SELECTED GRAIN SIZE AND CLAY MINERAL* PARAMETERS, BASED ON REPLICATE DATA FROM APPENDICES II, III, and IV.

Parameter	Source of Variation**	df	$\bar{X}$	s	Range
MUD (%)	Inter-station	38	54	27	2 -92
	Intra-station	15	52	5	15 -82
	Intra-sample	5	45	4	22 -89
MEAN SIZE (Ø)	Inter-station	38	4.5	1.4	1.2- 6.6
	Intra-station	15	4.5	0.3	2.6- 6.0
	Intra-sample	5	4.6	0.2	3.4- 6.9
EXPANDABLE MINERALS (%)	Inter-station	38	7	3.5	0 -14
	Intra-station	15	8	2	7 -12
	Intra-sample	3	13	2	11 -16
	Slide Mounting	29	9	1	2 -16
	Analysis and Calculations	18	9	1	4 -14
ILLITE (%)	Inter-station	38	62	4.4	54 -71
	Intra-station	15	62	4	58 -64
	Intra-sample	3	57	2	54 -59
	Slide Mounting	29	61	2	54 -68
	Analysis and Calculations	18	62	1	56 -70

* Clay mineral data are only for the less than 2μm sediment fraction.

** Statistics for each source calculated by pooling the variances of replicate data for multiple samples, except "inter-station" which represent one sample, with 39 measurements, of the Simpson Lagoon population.

APPENDIX X-B

STANDARD DEVIATIONS (s), WITH RELEVANT STATISTICS, OF EACH SOURCE OF VARIATION STUDIED FOR THREE CARBON PARAMETERS, BASED ON REPLICATE DATA FROM APPENDICES II and V

Parameter	Source of Variation*	df	$\bar{X}$	s	Range
TOTAL CARBON (%)	Intra-station	15	3	0.3	1 - 4
	Intra-subsample**	18	3	0.1	0.6- 5
	Intra-subsample***	9	3	0.06	0.4- 5
CARBONATE ($CO_3^{=}$) (%)	Inter-station	38	8	3	1 -13
	Intra-station	15	8	1	3 -12
	Intra-subsample	23	8	0.4	2 -14
ORGANIC CARBON (%)	Inter-station	38	1	0.9	0.1- 4.4
	Intra-station	15	1	0.2	0.3- 1.7

* Statistics for each source calculated by pooling the variances of replicate data for multiple samples, except "inter-station" which represents one sample, with 39 measurements, of the Simpson Lagoon population.

** Replicate subsamples analyzed on separate days with different instrument calibrations.

***Replicate subsamples analyzed on same day and calibration.

APPENDIX X-C

STANDARD DEVIATIONS (s), WITH RELEVANT STATISTICS, OF EACH SOURCE OF VARIATION STUDIED FOR TOTAL CONCENTRATIONS OF HEAVY METALS (μg/g, except Fe which is in 10^4 μg/g) IN SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDICES II and VI

Metal	Source of Variation*	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	38	72	20	29 -100
	Intra-station	15	70	6	40 - 96
	Intra-subsample	13	70	3	43 -100
	Aspiration	5	86	2	63 -107
CHROMIUM	Inter-station	38	47	15	16 - 70
	Intra-station	15	44	4	22 - 65
	Intra-subsample	15	43	2	21 - 66
	Aspiration	12	47	1	22 - 63
MANGANESE	Inter-station	38	270	108	115 -550
	Intra-station	15	250	37	130 -320
	Intra-subsample	13	200	3	120 -370
	Aspiration	2	530	7	510 -550
IRON	Inter-station	38	2.0	0.58	0.9- 2.9
	Intra-station	15	2.0	0.15	1.1- 2.7
	Intra-subsample	15	1.8	0.05	1.1- 2.8
	Aspiration	10	1.5	0.01	1.0- 2.3
COBALT	Inter-station	38	8	2	3 - 10
	Intra-station	15	7	1	4 - 9
	Intra-subsample	13	8	0.5	4 - 10
	Aspiration	7	6	0.4	3 - 10

APPENDIX X-C

(Continued)

Metal	Source of Variation*	df	$\bar{X}$	s	Range
NICKEL	Inter-station	38	23	7	8- 32
	Intra-station	15	22	1.6	10- 30
	Intra-subsample	14	22	0.8	10- 30
	Aspiration	12	25	0.7	10- 29
COPPER	Inter-station	38	17	6	6- 33
	Intra-station	15	16	3	7- 19
	Intra-subsample	11	15	2.4	8- 22
	Aspiration	2	13	0	6- 20
ZINC	Inter-station	38	75	23	28-120
	Intra-station	12	78	7	41- 98
	Intra-subsample	10	70	4	34-100
	Aspiration	12	78	3	37- 97

* Statistics for each source calculated by pooling the variances of replicate data for multiple samples, except "inter-station" which represents one sample, with 39 measurements, of the Simpson Lagoon population.

APPENDIX X-D

STANDARD DEVIATIONS (s), WITH RELEVANT STATISTICS, OF EACH SOURCE OF VARIATION STUDIED FOR EXTRACTABLE* CONCENTRATIONS OF HEAVY METALS (μg/g) IN SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDICES II AND VII

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	38	4	1.6	1 - 7
	Intra-station	15	4	0.4	2 - 6
	Intra-subsample	15	4	0.3	2 - 7
	Calibration	5	4	0.4	2 - 5
	Aspiration	35	4	0.3	1 - 6
CHROMIUM	Inter-station	38	1	0.6	0.0- 2.1
	Intra-station	15	1	0.2	0.5- 1.5
	Intra-subsample	12	1	0.1	0.2- 2.0
	Calibration	5	1	0.1	0.1- 1.5
	Aspiration	44	1	0.1	0.2- 2.3
MANGANESE	Inter-station	38	138	79	45 - 365
	Intra-station	15	124	28	55 - 175
	Intra-subsample	15	140	9	40 - 365
	Calibration	7	125	2	44 - 205
	Aspiration	4	257	1	140 - 360
IRON	Inter-station	38	2200	1030	300 -4700
	Intra-station	15	2100	300	1100 -2900
	Intra-subsample	14	2300	200	940 -4600
	Calibration	8	2500	46	800 -4600
	Aspiration	12	2300	36	940 -2800

APPENDIX X-D

(Continued)

Metal	Source of Variation**	df	$\bar{X}$	s	Range
COBALT	Inter-station	38	2	0.9	0.3- 3.5
	Intra-station	15	2	0.3	0.7- 2.9
	Intra-subsample	13	2	0.2	0.5- 3.6
	Calibration	5	2	0.2	0.7- 2.7
	Aspiration	51	2	0.1	0.4- 3.4
NICKEL	Inter-station	38	4	1.6	0.2- 6
	Intra-station	15	3	0.6	1.0- 5
	Intra-subsample	12	3	0.4	0.7- 5
	Calibration	8	3	0.2	0.7- 6
	Aspiration	45	4	0.1	0.7- 6
COPPER	Inter-station	38	3	0.8	0.4- 4.8
	Intra-station	14	3	0.3	1.7- 3.5
	Intra-subsample	16	3	0.5	1.7- 4.8
	Calibration	8	4	0.2	1.2- 8.9
	Aspiration	7	2	0.1	1.1- 3.3
ZINC	Inter-station	38	13	5.7	3 -23
	Intra-station	14	13	1.6	5 -20
	Intra-subsample	13	12	0.7	4 -20
	Calibration	13	14	0.5	4 -21
	Aspiration	22	12	0.3	3 -22

* Hydroxylamine hydrochloride-acetic acid reagent.

** Statistics for each source calculated by pooling the variances of replicate data for multiple samples, except "inter-station" which represents one sample, with 39 measurements, of the Simpson Lagoon population.

APPENDIX X-E

STANDARD DEVIATIONS (s), WITH RELEVANT STATISTICS, OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (µg/g) OF HEAVY METALS REMOVED BY STEP ONE* FROM FOUR SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDIX VIII-A

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	3	0.26	0.11	0.13- 0.36
	Intra-subsample	7	0.25	0.18	0.11- 0.74
CHROMIUM	Inter-station	3	0.067	0.050	0.02- 0.14
	Intra-subsample	7	0.068	0.092	0.01- 0.33
MANGANESE	Inter-station	3	21.3	11.6	9.4 -36.7
	Intra-subsample	9	21.4	4.9	8.5 -42.0
IRON	Inter-station	3	15.2	10.2	1.6 -26.2
	Intra-subsample	9	15.3	10.6	0.1 -41.0
COBALT	Inter-station	3	0.035	0.015	0.02- 0.05
	Intra-subsample	7	0.036	0.016	0.01- 0.07
NICKEL	Inter-station	3	0.186	0.085	0.06- 0.23
	Intra-subsample	7	0.182	0.035	0.02- 0.25
COPPER	Inter-station	3	0.54	0.24	0.23- 0.75
	Intra-subsample	7	0.55	0.17	0.18- 1.0

* See Table 5 in text.

** Inter-station variation statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-F

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS, OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS REMOVED BY STEP TWO* FROM FIVE SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDIX VIII-B

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	4	0.93	0.56	0.12- 1.55
	Intra-subsample	19	0.88	0.57	0.0 - 2.5
CHROMIUM	Inter-station	4	0.37	0.18	0.12- 0.61
	Intra-subsample	18	0.39	0.31	0.0 - 1.4
MANGANESE	Inter-station	4	45.4	24	20.8 - 71.7
	Intra-subsample	19	47.5	22	6 -130
IRON	Inter-station	4	194	107	38 -303
	Intra-subsample	19	177	97	0.0 -480
COBALT	Inter-station	4	0.42	0.18	0.16- 0.66
	Intra-subsample	19	0.42	0.27	0.0 - 1.4
NICKEL	Inter-station	4	2.4	1.5	0.46- 4.2
	Intra-subsample	19	2.2	1.4	0.0 - 6.3
COPPER	Inter-station	4	2.1	1.3	0.58- 3.9
	Intra-subsample	17	1.8	1.6	0.0 - 5.6
ZINC	Inter-station	4	8.5	3.8	2.5 - 12.0
	Intra-subsample	19	8.5	4.6	0.0 - 21

* See Table 5 in text.

** Inter-station variation statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-G

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS, OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS REMOVED BY STEP THREE* FROM SIX SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDIX VIII-C

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	5	0.45	0.33	0.18- 0.88
	Intra-subsample	19	0.44	0.45	0.0 - 1.9
CHROMIUM	Inter-station	5	0.059	0.040	0.03- 0.13
	Intra-subsample	19	0.066	0.074	0.00- 0.27
MANGANESE	Inter-station	5	19.0	22.1	0.53- 60
	Intra-subsample	19	16.5	25.5	0.0 - 120
IRON	Inter-station	5	487	336	169 -1069
	Intra-subsample	19	478	401	34 -2150
COBALT	Inter-station	5	0.194	0.085	0.11- 0.34
	Intra-subsample	19	0.181	0.200	0.02- 0.71
NICKEL	Inter-station	5	0.036	0.027	0.00- 0.06
	Intra-subsample	19	0.039	0.057	0.0 - 0.16
COPPER	Inter-station	5	3.0	1.3	1.09- 4.67
	Intra-subsample	19	2.9	2.2	0.17- 9.5

* See Table 5 in text.

** Inter-station variation statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-H

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS, OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS REMOVED BY STEP FOUR* FROM SIX SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDIX VIII-D

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	5	0.76	0.63	0.23- 1.8
	Intra-subsample	22	0.61	0.70	0.08- 4.1
CHROMIUM	Inter-station	5	0.063	0.038	0.03- 0.13
	Intra-subsample	22	0.060	0.084	0.0 - 0.36
MANGANESE	Inter-station	5	19.0	9.3	11.5 - 36.6
	Intra-subsample	22	19.5	17.0	3 - 68
IRON	Inter-station	5	152	74	85 -246
	Intra-subsample	22	151	131	8 -510
COBALT	Inter-station	5	0.20	0.08	0.09- 0.30
	Intra-subsample	22	0.21	0.17	0.02- 0.67
NICKEL	Inter-station	5	0.41	0.24	0.18- 0.68
	Intra-subsample	22	0.41	0.44	0.03- 1.5
COPPER	Inter-station	5	0.19	0.10	0.05- 0.34
	Intra-subsample	22	0.20	0.22	0.0 - 0.79
ZINC	Inter-station	5	2.1	0.9	1.05- 3.40
	Intra-subsample	22	2.2	1.3	0.60- 5.40

* See Table 5 in text.

** Inter-station variance statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-I

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS, OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS REMOVED BY STEP FIVE* FROM SIX SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDIX VIII-E

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	5	3.7	1.3	1.6 - 4.9
	Intra-subsample	18	3.5	1.2	0.5 - 6.1
CHROMIUM	Inter-station	5	0.80	0.32	0.16- 1.0
	Intra-subsample	18	0.74	0.39	0.07- 1.5
MANGANESE	Inter-station	5	14.2	9.2	5.1 - 24.6
	Intra-subsample	18	13.8	12.9	0.1 - 42
IRON	Inter-station	5	3520	1290	1120 -4600
	Intra-subsample	18	3290	875	670 -6120
COBALT	Inter-station	5	0.65	0.37	0.18- 1.2
	Intra-subsample	17	0.59	0.49	0.03- 1.8
NICKEL	Inter-station	5	2.1	1.0	0.42- 3.4
	Intra-subsample	17	1.8	1.0	0.16- 5.3
COPPER	Inter-station	5	3.9	1.7	1.0 - 5.7
	Intra-subsample	18	3.6	1.8	0.5 - 9.3
ZINC	Inter-station	5	5.9	2.8	1.9 - 10.1
	Intra-subsample	18	5.5	6.0	0.0 - 17.5

* See Table 5 in text.

** Inter-station variance statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-J

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS, OF INTER-STATION AND INTRA-SUBSAMPLE SOURCE OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS REMOVED BY STEP SIX* FROM SIX SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDIX VIII-F

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	5	3.0	1.2	1.3 - 4.3
	Intra-subsample	20	2.9	0.5	1.0 - 5.0
CHROMIUM	Inter-station	5	0.84	0.40	0.33- 1.44
	Intra-subsample	20	0.83	0.75	0.0 - 3.0
MANGANESE	Inter-station	5	27.8	14.6	8.7 - 41.8
	Intra-subsample	20	27.6	3.3	7 - 50.5
IRON	Inter-station	5	3540	1440	1500 -4920
	Intra-subsample	20	3560	310	1280 -5420
COBALT	Inter-station	5	1.63	0.43	0.81- 2.0
	Intra-subsample	20	1.55	0.33	0.64- 2.2
NICKEL	Inter-station	5	2.48	1.12	0.76- 3.5
	Intra-subsample	20	2.39	0.75	0.53- 4.4
COPPER	Inter-station	5	1.04	0.46	0.29- 1.5
	Intra-subsample	20	1.01	0.42	0.0 - 2.2

* See Table 5 in text.

** Inter-station variance statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-K

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS, OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS REMOVED BY STEP SEVEN* FROM SIX SIMPSON LAGOON SEDIMENTS, BASED ON REPLICATE DATA FROM APPENDIX VIII-G

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	5	2.34	1.22	0.70- 4.5
	Intra-subsample	20	2.20	0.66	0.35- 5.6
CHROMIUM	Inter-station	5	2.2	1.16	0.31- 3.9
	Intra-subsample	20	2.1	0.45	0.12- 4.6
MANGANESE	Inter-station	5	20.2	11.1	5.3 - 36.5
	Intra-subsample	20	18.7	6.5	3.9 - 50
IRON	Inter-station	5	2100	1010	503 -3490
	Intra-subsample	19	1980	470	395 -4000
COBALT	Inter-station	5	0.92	0.40	0.3 - 1.4
	Intra-subsample	20	0.85	0.45	0.0 - 2.0
NICKEL	Inter-station	5	3.5	1.44	1.1 - 5.5
	Intra-subsample	20	3.3	0.64	0.6 - 6.9
COPPER	Inter-station	5	2.8	1.0	1.0 - 3.7
	Intra-subsample	20	2.6	1.2	0.7 - 6.5

* See Table 5 in text.

** Inter-station variance statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-L

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS IN FIVE SIMPSON LAGOON SEDIMENTS EXTRACTED INTO STEPS (1-4)* OF THE EIGHT-STEP SEQUENCE, BASED ON REPLICATE DATA FROM APPENDICES IX-A to -F

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	4	2.5	1.5	0.72- 4.7
	Intra-subsample	20	2.2	0.7	0.23- 6.1
CHROMIUM	Inter-station	4	0.71	0.39	0.26- 1.1
	Intra-subsample	16	0.71	0.31	0.09- 1.6
MANGANESE	Inter-station	4	101	58	57 - 190
	Intra-subsample	18	96	19	27 - 218
IRON	Inter-station	4	740	282	347 -1100
	Intra-subsample	15	668	317	165 -1660
COBALT	Inter-station	4	0.88	0.26	0.50- 1.2
	Intra-subsample	18	0.87	0.31	0.32- 1.8
NICKEL	Inter-station	4	3.3	1.9	0.73- 5.7
	Intra-subsample	18	3.0	1.7	0.18- 9.1
COPPER	Inter-station	4	5.3	1.9	2.3 - 7.6
	Intra-subsample	17	4.8	2.4	1.0 - 10.8

* Extraction Group I: Leachable Fractions.

** Inter-station variation statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-M

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS IN SIX SIMPSON LAGOON SEDIMENTS EXTRACTED INTO STEPS (5-7)* OF THE EIGHT-STEP SEQUENCE, BASED ON REPLICATE DATA FROM APPENDICES IX-A to -F

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	5	9.2	2.9	4.2 - 12.2
	Intra-subsample	20	8.8	1.4	2.2 - 13.8
CHROMIUM	Inter-station	5	4.1	1.8	0.9 - 6.0
	Intra-subsample	20	4.0	0.9	0.6 - 7.3
MANGANESE	Inter-station	5	61	32	23 - 94
	Intra-subsample	20	59	10	18 - 121
IRON	Inter-station	5	9170	3330	3710 -12550
	Intra-subsample	18	8740	1040	3030 -13920
COBALT	Inter-station	5	3.1	1.1	1.3 - 4.3
	Intra-subsample	18	2.9	0.6	1.0 - 4.8
NICKEL	Inter-station	5	8.0	3.1	2.2 - 11.1
	Intra-subsample	18	7.3	0.6	1.4 - 11.5
COPPER	Inter-station	5	7.9	3.0	2.5 - 11.3
	Intra-subsample	20	7.4	2.4	1.5 - 14.7

* Extraction Group II: Iron Phases.

** Inter-station variation statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX X-N

STANDARD DEVIATIONS (s) WITH RELEVANT STATISTICS, OF INTER-STATION AND INTRA-SUBSAMPLE SOURCES OF VARIATION IN CONCENTRATIONS (μg/g) OF HEAVY METALS IN FIVE SIMPSON LAGOON SEDIMENTS EXTRACTED INTO STEPS (1-7)* OF THE EIGHT-STEP SEQUENCE, BASED ON REPLICATE DATA FROM APPENDICES IX-A to -F

Metal	Source of Variation**	df	$\bar{X}$	s	Range
VANADIUM	Inter-station	4	11.8	4.3	4.9 - 16.4
	Intra-subsample	18	11.0	1.9	3.0 - 19.9
CHROMIUM	Inter-station	4	4.9	2.3	1.3 - 7.2
	Intra-subsample	16	4.8	1.0	1.1 - 7.9
MANGANESE	Inter-station	4	168	80	80 - 285
	Intra-subsample	17	158	23	49 - 297
IRON	Inter-station	4	10250	3960	4050 -13600
	Intra-subsample	14	9290	716	3580 -14500
COBALT	Inter-station	4	3.9	1.3	1.8 - 5.1
	Intra-subsample	16	3.6	0.7	1.4 - 5.8
NICKEL	Inter-station	4	11.0	4.7	3.0 - 15.2
	Intra-subsample	14	9.6	1.7	1.8 - 18.8
COPPER	Inter-station	4	12.9	5.4	4.8 - 19.5
	Intra-subsample	16	11.7	2.9	3.8 - 21.3

* Sum of Extraction Groups I and II.

** Inter-station variation statistics calculated with the mean values of replicates for each station. Statistics for intra-subsample variations calculated by pooling the variances of replicate data for each station.

APPENDIX XI

TOTAL HEAVY-METAL CONTENTS OF SEDIMENTS FROM TWO STATIONS (2, 23) OF SIMPSON LAGOON, MEASURED BY SUMMING THE METALS REMOVED BY EACH STEP IN FOUR EXTRACTION SEQUENCES, AND BY THE TOTAL DIGESTION OF A WHOLE SUBSAMPLE

APPENDIX XI-A

TOTAL SEDIMENT CONCENTRATIONS (µg/g) OF HEAVY METALS* IN FIVE SUBSAMPLES OF SL877-2, DETERMINED FROM THE SUM OF METALS EXTRACTED BY FOUR SEQUENCES AND THE HYDROFLUORIC-NITRIC ACID (HFNA) DIGESTION OF A WHOLE SUBSAMPLE

Step**		Sequence Four***	Seven†	Eight‡	Nine◇	HFNA Digest☆	s△
				CHROMIUM			
1-7	SUM	1.3	0.7	1.4	0.7		
8		14.8	16.2	18.5	15.8		
	TOTAL	16.1	16.9	19.9	16.5	21	2
				MANGANESE			
1-7	SUM	49	89	101	96		
8		35	50	35	35		
	TOTAL	84	139	136	131	146	3
				IRON			
1-7	SUM	3880	2140	5538	4560		
8		4940	8010◆	5120	4990		
	TOTAL	8820	10150	10660	9550	9500	450
				NICKEL			
1-7	SUM	2.3	2.2	3.5	2.5		
8		2.4★	2.4★	2.4★	4.8★		
	TOTAL	4.7	4.6	5.9	7.3	11	0.8
				COPPER			
1-7	SUM	5.5	2.4	3.4	2.5		
8		4.7	6.0	4.1	4.2		
	TOTAL	10.2	8.4	7.5	6.7	11	2.4

APPENDIX XI-A

(Continued)

Step**		Sequence Four***	Seven†	Eight‡	Nine◇	HFNA Digest☆	s△
			ZINC				
1-7	SUM	I▲	8.5	I▲	I▲		
8		10.7	27◆	13.4	10.7		
	TOTAL		35.5			28	4

* V, Co not analyzed in Step 8 extracts.
** Refer to Table 5 for explanation.
*** From Appendix IX-A.
† Steps 2-6 omitted.
‡ Steps 1-3 omitted.
◇ Steps 1-5 omitted.
☆ From Appendices II-D,E.
△ From Appendix X-C.
◆ Omitted from average calculations for Appendix XII-A.
★ Represents less than 1% absorption.
▲ Incomplete sequence due to zinc contamination in some steps.

APPENDIX XI-B

TOTAL SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS* IN FIVE SUBSAMPLES OF SL877-23, DETERMINED FROM THE SUM OF METALS EXTRACTED BY FOUR SEQUENCES AND THE HYDROFLUORIC-NITRIC ACID (HFNA) DIGESTION OF A WHOLE SUBSAMPLE

Step**		Sequence				HFNA	
		Four***	Seven†	Eight‡	Nine◇	Digest☆	s△
				CHROMIUM			
1-7	SUM	4.3	3.8	7.2	5.5		
8		60	61	60	59		
	TOTAL	64.3	64.8	67.2	64.5	56	2
				MANGANESE			
1-7	SUM	176	230	218	254		
8		85	134	75	79		
	TOTAL	261	364	293	333	380	3
				IRON			
1-7	SUM	11940	6120	14170	13240		
8		14100	22300◆	12600	13600		
	TOTAL	26040	28420	26770	26840	20900	450
				NICKEL			
1-7	SUM	9.0	8.7	12.5	11.4		
8		16.8	18.9	14.2	14.2		
	TOTAL	25.8	27.6	26.7	25.6	27	0.8
				COPPER			
1-7	SUM	13.0	13.0	14.1	14.6		
8		5.8	10.4◆	5.7	5.2		
	TOTAL	18.8	23.4	19.8	19.8	21	2.4

APPENDIX XI-B

(Continued)

Step**		Sequence Four***	Seven†	Eight‡	Nine◇	HFNA Digest☆	s△
				ZINC			
1-7	SUM	I★	33	I★	I★		
8		38	61◆	32	40		
	TOTAL		94			93	4

* V, Co not analyzed in Step 8 extracts.
** Refer to Table 5 for explanation.
*** From Appendix IX-D.
† Steps 2-6 omitted.
‡ Steps 1-3 omitted.
◇ Steps 1-5 omitted.
☆ From Appendices II-D,E.
△ From Appendix X-C.
◆ Omitted from average calculations for Appendix XII-E.
★ Incomplete sequence due to zinc contamination in some steps.

APPENDIX XII

AVERAGES FOR THE PARTITIONING OF HEAVY METALS AMONG CHEMICAL FRACTIONS (extraction steps) IN SEDIMENTS FROM SIX STATIONS OF SIMPSON LAGOON

APPENDIX XII-A

AVERAGE SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS PARTITIONED BY CHEMICAL EXTRACTIONS FOR THE SAND SL877-2

Method of Partitioning	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
BY STEP*								
Step 1	0.13	0.02	9	2	0.02	0.06	0.25	
Step 2	0.1	0.1	20	40	0.2	0.5	0.6	3
Step 3	0.2	0.03	20	200	0.15	0.03	1	
Step 4	0.2	0.03	15	100	0.2	0.2	0.35	2
Group I SUM	0.63	0.18	64	342	0.57	0.79	2.2	>5
Step 5	1.5	0.15	5	1100	0.2	0.4	1	2
Step 6	2	0.3	12	2100	0.8	0.8	0.3	
Step 7	0.7	0.3	5	500	0.3	1	1	
Group II SUM	4.2	0.75	22	3700	1.3	2.2	2.3	
Steps 1-7 SUM	4.8	0.93	86	4040	1.9	3.0	4.5	>7
Step 8**	-	16.3	39	5020	-	3.0	4.8	11.6
Eight Step Total	-	17.2	125	9060	-	6.0	9.3	
BY SEQUENCE***								
Group I	0.7	0.25	57	350	0.5	0.7	2.3	
Group II	4.2	0.9	23	3700	1.3	2.2	2.5	
I + II	4.9	1.3	80	4050	1.8	3.0	4.8	
HHAA EXTRACTION†	2	0.4	80	1100	0.7	1	1.1	3
HFNA DIGEST†	36	21	146	9500	3.6	11	11	28
APPENDIX XI-A TOTAL	-	17.4	123	9800	-	5.6	8.2	36‡

* Taken from Appendix VIII.
** Taken from Appendix XI-A.
*** Taken from Appendix IX-A.
† Taken from Appendices II-D,E.
‡ Single value.

APPENDIX XII-B

AVERAGE SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS PARTITIONED BY CHEMICAL EXTRACTIONS FOR THE SILT SL877-13

Method of Partitioning	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
BY STEP*								
Step 1	0.2	0.06	35	15	0.05	0.25	0.75	
Step 2	0.6	0.35	70	140	0.35	2	2	9
Step 3	0.2	0.03	60	400	0.35	0.01	3.5	
Step 4	0.5	0.05	35	85	0.15	0.2	0.2	1.3
Group I SUM	1.5	0.49	200	640	0.9	2.46	6.45	>10
Step 5	4	1	25	4600	0.8	2	4.5	7
Step 6	3.7	1	40	4100	2	3.5	1.5	
Step 7	4.5	4	35	3500	1.5	5.5	3	
Group II SUM	12.2	6	100	12200	4.3	11	9	
Steps 1-7 SUM	13.7	6.5	300	12800	5.1	13.5	15.5	>17
BY SEQUENCE**								
Group I	1.6	0.5	190	820	1	2.2	5.6	
Group II	12.2	6	97	12500	3.9	10.3	9	
I + II	13.8	7.2	285	13500	4.9	13	13.7	
HHAA EXTRACTION***	5.1	1.8	300	3700	3	5	3.5	18
HFNA DIGEST***	79	60	467	26400	9.2	29	23	88

* Taken from Appendix VIII.
** Taken from Appendix IX-B.
*** Taken from Appendices II-D,E.

APPENDIX XII-C

ESTIMATE OF STEP-ONE AVERAGE FOR SL877-18 TO BE USED IN APPENDIX XII-D, BASED ON AVERAGES FOR OTHER SEDIMENTS

Station	Step-One Average*						
	V	Cr	Mn	Fe	Co	Ni	Cu
2	0.13	0.02	9	2	0.02	0.06	0.25
13	0.2	0.06	35	15	0.05	0.25	0.75
23	0.35	0.15	25	15	0.04	0.25	0.7
28	0.35	0.07	9	7	0.03	0.25	0.7
UG- 1	0.35	0.05	17	25	0.03	0.23	0.47
$\bar{X}$	0.28	0.07	19	13	0.03	0.21	0.57
Use for Step One in Appendix XII-D:	0.3	0.08	20	15	0.03	0.2	0.6

* Taken from Appendix XII.

APPENDIX XII-D

AVERAGED SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS PARTITIONED BY CHEMICAL EXTRACTIONS FOR THE PEATY, SILTY SAND SL877-18

Method of Partitioning	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
BY STEP*								
Step 1**	0.3	0.08	20	15	0.03	0.2	0.6	
Step 2***	3.7	0.14	16	660	1.2	2.5	3.4	10.5
Step 3	0.9	0.07	0.5	1100	0.3	0.06	5	
Step 4	1.3	0.15	12	250	0.3	0.7	0.2	2.5
Group I SUM	6.2	0.44	48.5	2025	1.83	3.46	9.2	>13
Step 5	5	0.9	7	4200	0.7	3	4.5	5.5
Step 6	1.3	0.5	9	1500	1.5	1.5	0.7	
Step 7	2	2	16	2100	1.3	3.5	3	
Group II SUM	8.3	3.4	32	7800	3.5	8	8.2	
Steps 1-7 SUM	14.5	3.8	80	9825	5.3	11.5	17.4	>19
BY SEQUENCE†								
Group II‡	8.6	4.1	32	7800	3.6	7.9	8.6	
HHAA EXTRACTION◇	6.7	1.7	60	4100	3.1	6.1	2.3	22
HFNA DIGEST◇	74	54	163	19300	8.7	27	20	78

* Taken from Appendix VIII.
** Estimate from Appendix XII-C.
*** Only one value.
† Taken from Appendix IX-C.
‡ Group I data incomplete.
◇ Taken from Appendices II-D,E.

APPENDIX XII-E

AVERAGE SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS PARTITIONED BY CHEMICAL EXTRACTIONS FOR THE SANDY SILT SL877-23

Method of Partitioning	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
BY STEP*								
Step 1	0.35	0.15	25	15	0.04	0.25	0.7	
Step 2	1.5	0.6	70	270	0.7	2	1.5	11
Step 3	0.35	0.15	25	400	0.2	0.06	3.5	
Step 4	0.35	0.09	20	230	0.3	0.6	0.2	3.5
Group I SUM	2.55	0.99	140	915	1.24	2.91	5.9	>15
Step 5	4	1	20	4100	0.6	2	5	7
Step 6	2.9	0.9	39	4900	1.7	3	1.5	
Step 7	2.1	2.4	20	2200	0.8	3.5	3	
Group II SUM	9	4.3	79	11200	3.1	8.5	9.5	
Steps 1-7 SUM	11.6	5.3	220	12100	4.3	11.4	15.4	>22
Step 8**	-	60	93	13400	-	16.0	5.6	37
Eight Step Total	-	65.3	313	25500	-	27.4	21.0	
BY SEQUENCE***								
Group I	2.8	1.1	128	840	1.2	3.3	5.9	
Group II	9.7	4.8	77	11170	2.9	8.5	9.2	
I + II	12.4	6	205	12100	3.9	11.8	15.1	
HHAA EXTRACTION†	4.4	1.2	215	2480	2.7	4.7	2.7	15
HFNA DIGEST†	70	56	380	20900	8.4	27	21	93
APPENDIX XI-B TOTAL	-	65.2	313	27000	-	26.4	20.5	94‡

* Taken from Appendix VIII.
** Taken from Appendix XI-B.
*** Taken from Appendix IX-D.
† Taken from Appendices II-D,E.
‡ Single value.

APPENDIX XII-F

AVERAGE SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS PARTITIONED BY CHEMICAL EXTRACTIONS FOR THE CLAYEY SILT SL877-28

Method of Partitioning	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
BY STEP*								
Step 1	0.35	0.07	9	7	0.03	0.25	0.7	
Step 2	1.3	0.3	29	300	0.45	3.5	4	8
Step 3	0.9	0.06	5	680	0.15	0.002	3.9	
Step 4	2	0.03	20	85	0.09	0.2	0.15	1.1
Group I SUM	4.55	0.46	63	1072	0.72	3.95	8.75	>9
Step 5	5	0.9	25	4100	1.5	3.5	5.5	10
Step 6	4.3	0.7	40	4900	2.0	3.5	1.2	
Step 7	2.5	2.0	30	2700	1.0	4	3.5	
Group II SUM	11.8	3.6	95	11700	4.5	11	10.2	
Steps 1-7 SUM	16.4	4.1	158	12800	5.2	15	19	>19
BY SEQUENCE**								
Group I	4.7	0.5	60	1100	0.8	4	8	
Group II	11.7	3.7	94	11800	4.3	11.1	11.3	
I + II	16.4	4.2	154	12900	5.1	15.2	19.5	
HHAA EXTRACTION***	5.1	1.8	114	2740	2.3	4.3	2.8	16
HFNA DIGEST***	93	62	290	26700	9.3	32	23	100

* Taken from Appendix VIII.
** Taken from Appendix IX-E.
*** Taken from Appendices II-D,E.

APPENDIX XII-G

AVERAGE SEDIMENT CONCENTRATIONS (μg/g) OF HEAVY METALS PARTITIONED BY CHEMICAL EXTRACTIONS FOR THE PEATY, CLAYEY-SILT SAND UG-1

Method of Partitioning	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
BY STEP*								
Step 1	0.35	0.05	17	25	0.03	0.23	0.47	
Step 2	1	0.4	35	220	0.5	4	2.5	12
Step 3	0.25	0.04	5	170	0.1	0.06	2	
Step 4	0.4	0.05	12	170	0.2	0.6	0.05	2.5
Group I SUM	2.0	0.54	69	585	0.83	4.89	5.02	>15
Step 5	2.8	0.8	6	3000	0.4	2	3	4
Step 6	4	1.5	26	3750	1.7	2.5	1.2	
Step 7	2.3	2.5	15	1600	0.7	3.5	2	
Group II SUM	9.1	4.8	47	8350	2.8	8	6.2	
Steps 1-7 SUM	11.1	5.3	116	8935	3.6	12.9	11.2	>19
BY SEQUENCE**								
Group I	2.4	1.1	70	600	0.89	5.7	5.0	
Group II	9.1	5.4	47	8033	2.8	7.8	6.8	
I + II	11.4	6.1	116	8600	3.7	11.9	11.5	
HHAA EXTRACTION***	3.5	0.7	90	1500	2.1	4	0.4	12
HFNA DIGEST***	56	46	211	16500	6.0	21	16	65

* Taken from Appendix VIII.
** Taken from Appendix IX-F.
*** Taken from Appendices II-D,E.

APPENDIX XIII

DETAILS ABOUT THE ANOVA RUN ON THE AMOUNTS OF EXTRACTABLE METALS (Groups I & II in Appendix IX) IN SEDIMENTS FROM SIX STATIONS OF SIMPSON LAGOON

The precision was poor for the amounts of heavy metals extracted from six sediments of Simpson Lagoon with the multi-step scheme. As such, it did not seem justified to discriminate between the six stations using the data from single treatments of the eight-step scheme. Discrimination between stations was made instead with the more precise data produced by combining extraction steps (Groups I & II). In order to determine if there is a significant difference between the station means of the extractable metals in Groups I & II, a one-way analysis of variance (ANOVA) was undertaken. This test compares, for metals removed by the treatments of each extraction group, the variance between means of replicates from each station (inter-station variance), with the variance of replicates within subsamples from each station (intra-subsample variance).

It is to be noted that the inter-station variance was calculated using the mean values of replicates run for each station. The resulting variance between station means must be multiplied by the number of replicates (n)

run in each station, in order that the variability within the entire population be correctly estimated (Freund, 1979, p. 414). In this study, the number of replicates run for each station were different, making it necessary to use a specific average number: the harmonic mean (n_h; Snedecor & Cochran, 1980, pp. 229, 418; and outlined in the footnotes of Tables 11, 12). The station means used in the ANOVA are listed in Appendix XII and Appendix XIV ($\bar{X}_i$).

The intra-subsample estimate of the population variance is obtained by pooling the variances between subsamples from each station. Data used are listed in Appendix IX. Again, the number of replicates for each station are not equal. Also, the intra-subsample variances for each station are assumed not to be equal--the normal assumption for an ANOVA: i.e., the estimated variances between subsamples from each station appear dissimilar (Appendices XIV-A,B). Appendices XIV-A,B in part summarize the estimates of the intra-subsample variances calculated from the data in Appendix IX, for each of the six stations with sample replicates subjected to the sequential extractions. The individual variances thus calculated for each station were pooled together before being listed in Appendices X-L and X-M. As seen

in Appendices XIV-A,B, replicate data for the sandy sediment from station 2 usually have a much smaller variation (s_i^2) relative to the other stations. This may possibly be due to the smaller means of the replicates ($\bar{X}_i$) for station 2. Additionally, only two replicates, with very different values, were analyzed for some stations (Appendix IX). The variance among replicates for these stations were thus unusually large compared to the variance among replicates for the other stations. So, because of a contrast in sample means, and poorer reproducibility for some replicates, it was not assumed that intra-subsample variances from each station were equal.

The procedure used to compensate for an inequality of variances among pooled samples is an extension of Satterthwaite's approximation (Snedecor & Cochran, 1980, p. 97). Satterthwaite's approximation assigns an approximate number of degrees of freedom (df) so that student's t tables, which assume equal variances, can be used to compare two sample means. These calculated df's (Appendices XIV-A,B; Tables 11,12) are smaller than the total df's for the subsample replicates shown in Appendices X-L,M. For the F-test performed in this study, the estimate of the df of the intra-subsample variance data follows the procedure of Example 12.10.1 on page 229 in

Snedecor & Cochran (1980). Results of these calculations are shown in Appendices XIV-A,B.

Therefore, the F statistic to test the differences between the means of the six stations having heavy-metal data for Extraction Groups I and II, is the ratio of two estimates for the population variance: 1) inter-station variance between station means, for an unequal number of replicates at each station (used n_h); 2) pooled intra-subsample variances, using an approximate df to compensate for unequal variances among replicates at each station.

APPENDIX XIV

CALCULATIONS OF THE APPROXIMATE DEGREES OF FREEDOM FOR THE POOLED ESTIMATES OF INTRA-SUBSAMPLE VARIANCE USED IN THE ANOVA PRESENTED IN TABLES 11 & 12

APPENDIX XIV-A

CALCULATIONS OF THE APPROXIMATE[1] DEGREES OF FREEDOM (df_{uv}) FOR THE POOLED[2] ESTIMATES OF INTRA-SUBSAMPLE VARIANCE USED IN THE ANOVA ON THE AMOUNTS OF HEAVY METALS EXTRACTED BY GROUP I TREATMENTS[3]

Station	$\bar{X}_i$	df_i	s_i^2	$df_i s_i^2$	$df_i (s_i^2)^2$	df_{uv} #
			V A N A D I U M			
2	0.72	5	0.08	0.40	0.03	
13	1.59	4	0.34	1.36	0.46	
23	2.78	5	0.58	2.91	1.68	
28	4.73	2	1.52	3.05	4.62	
UG- 1	2.42	4	0.67	2.67	1.80	
				$\Sigma_1 = 10.4$	$\Sigma_2 = 8.6$	13
			C H R O M I U M			
2	0.26	4	0.01	0.06	0.00[a]	
13	0.49	3	0.04	0.11	0.00[a]	
23	1.14	5	0.15	0.77	0.12[a]	
28	0.54	2	0.26	0.05	0.00[a]	
UG- 1	1.10	2	0.27	0.54	0.14	
				$\Sigma_1 = 1.5$	$\Sigma_2 = 0.27$	9
			M A N G A N E S E			
2	57	5	443	2215	981[b]	
13	190	2	602	1205	725[b]	
23	128	5	566	2831	1602[b]	
28	60	2	145	291	42[b]	
UG- 1	70	4	13	53	1*	
				$\Sigma_1 = 6595$	$\Sigma_2 = 3350$[b]	13

APPENDIX XIV-A

(Continued)

Station	$\bar{X}_i$	df_i	s_i^2	$df_i s_i^2$	$df_i (s_i^2)^2$	df_{uv} #
			I R O N			
2	347	5	39^b	195^b	8^c	
13	820	1	583^b	583^b	340^c	
23	837	3	24^b	72^b	2^c	
28	1097	2	313^b	627^b	197^c	
UG- 1	599	4	8^b	31^b	0^{**}	
				$\Sigma_1 = 1509^b$	$\Sigma_2 = 546^c$	4
			C O B A L T			
2	0.50	5	0.03	0.16	5.1^d	
13	1.03	2	0.05	0.09	4.4^d	
23	1.19	5	0.22	1.11	242.0^d	
28	0.81	2	0.07	0.15	10.6^d	
UG- 1	0.89	4	0.05	0.18	8.5^d	
				$\Sigma_1 = 1.7$	$\Sigma_2 = 0.27$	11
			N I C K E L			
2	0.7	5	0.3	1.3	0.3	
13	2.2	4	1.2	4.9	5.8	
23	3.3	4	0.3	1.2	0.4	
28	4.4	1	18.6	18.6	346.0	
UG- 1	5.7	4	6.3	25.3	159.8	
				$\Sigma_1 = 51.3$	$\Sigma_2 = 512$	5

APPENDIX XIV-A

(Continued)

Station	$\bar{X}_i$	df_i	s_i^2	$df_i s_i^2$	$df_i (s_i^2)^2$	df_{uv} #
			C O P P E R			
2	2.3	5	0.9	4.7	4.4	
13	5.6	2	0.9	1.8	1.7	
23	5.9	5	10.4	52.1	540.8	
28	7.6	1	20.5	20.5	419.4	
UG- 1	5.0	4	5.5	22.1	121.0	
				$\Sigma_1 = 101$	$\Sigma_2 = 1087$	9

[1] A detailed explanation is provided in Appendix XIII.

[2] Intra-subsample variances for each station (s_i^2) are assumed to be unequal.

[3] This ANOVA presented in Table 11.

$df_{uv} = (\Sigma_1)^2/\Sigma_2$, where "uv" denotes unequal variances.

[a] Number is < 0.005.

[b] Multiply by 10^3.

[c] Multiply by 10^9.

* Number = 676.

** Number = 2.4×10^8.

[d] Multiply by 10^{-3}.

APPENDIX XIV-B

CALCULATIONS OF THE APPROXIMATE[1] DEGREES OF FREEDOM (df_{uv}) FOR THE POOLED[2] ESTIMATES OF INTRA-SUBSAMPLE VARIANCE USED IN THE ANOVA ON THE AMOUNTS OF HEAVY METALS EXTRACTED BY GROUP II TREATMENTS[3]

Station	$\bar{X}_i$	df_i	s_i^2	$df_i s_i^2$	$df_i (s_i^2)^2$	df_{uv} #
			V A N A D I U M			
2	4.2	5	1.5	7.6	11.3	
13	12.2	3	2.0	6.0	12.0	
18	8.6	2	0.8	1.6	1.3	
23	9.7	5	1.8	9.2	16.9	
28	11.7	2	6.0	11.9	71.3	
UG- 1	9.1	3	1.8	5.2	9.2	
				Σ_1=41.6	Σ_2=122	14
			C H R O M I U M			
2	0.9	5	0.07	0.33	0.02	
13	6.0	3	1.33	3.98	5.31	
18	4.1	2	0.22	0.45	0.10	
23	4.8	5	1.44	7.18	10.37	
28	3.7	2	0.14	0.29	0.04	
UG- 1	5.4	3	1.54	4.63	7.11	
				Σ_1=16.9	Σ_2=22.9	12
			M A N G A N E S E			
2	22.8	5	11	55	605	
13	94.3	3	410	1229	503[a]	
18	31.7	2	26	53	1352	
23	76.8	5	88	441	39[a]	
28	94.0	2	117	234	27[a]	
UG- 1	47.3	3	33	99	3267	
				Σ_1=2110	Σ_2=575[a]	8

APPENDIX XIV-B

(Continued)

Station	$\bar{X}_i$	df_i	s_i^2	$df_i s_i^2$	$df_i (s_i^2)^2$	df_{uv} #
			I R O N			
2	3.7^a	5	257^a	1285^a	330^b	
13	12.5^a	2	164^a	327^a	54^b	
18	7.8^a	2	4130^a	8261^a	34122^b	
23	11.2^a	5	441^a	2204^a	972^b	
28	11.8^a	2	3551^a	7102^a	25218^b	
UG- 1	8.0^a	2	107^a	214^a	23^b	
				$\Sigma_1 = 19393^a$	$\Sigma_2 = 60719^b$	6
			C O B A L T			
2	1.3	5	0.12	0.59	0.07	
13	3.9	2	0.34	0.67	0.23	
18	3.6	2	0.93	1.86	1.73	
23	2.9	4	0.13	0.52	0.07	
28	4.3	2	0.61	1.22	0.74	
UG- 1	2.8	3	0.37	1.10	0.41	
				$\Sigma_1 = 6.0$	$\Sigma_2 = 3.2$	11
			N I C K E L			
2	2.2	5	0.31	1.53	0.47	
13	10.3	2	0.06	0.13	0.01	
18	7.9	2	0.01	0.03	0.00	
23	8.5	4	0.74	2.95	2.19	
28	11.1	2	0.44	0.89	0.39	
UG- 1	7.8	3	0.22	0.67	0.15	
				$\Sigma_1 = 6.2$	$\Sigma_2 = 3.2$	12

APPENDIX XIV-B

(Continued)

Station	$\bar{X}_i$	df_i	s_i^2	$df_i s_i^2$	$df_i (s_i^2)^2$	df_{uv} #
			C O P P E R			
2	2.5	5	0.3	1.5	0.4	
13	9.0	3	5.2	15.6	81.1	
18	8.6	2	21.6	43.2	933.1	
23	9.3	5	5.5	27.6	151.3	
28	11.3	2	9.0	18.1	162.0	
UG- 1	6.8	3	2.1	6.3	13.2	
				$\Sigma_1 = 112$	$\Sigma_2 = 1341$	9

[1] A detailed explanation is provided in Appendix XIII.

[2] Intra-subsample variances for each station (s_i^2) are assumed to be unequal.

[3] This ANOVA presented in Table 12.

$df_{uv} = (\Sigma_1)^2/\Sigma_2$, where "uv" denotes unequal variances.

[a] Multiply by 10^3.

[b] Multiply by 10^9.

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